INVESTIGATION INTO SPONTANEOUS COMBUSTION CHARACTERISTICS OF SOME INDIAN COALS AND CORRELATION STUDY WITH THEIR INTRINSIC PROPERTIES

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology

In

Mining Engineering

By

SHIVA SHANKAR JENA
107MN010

DEPARTMENT OF MINING ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY
ROURKELA-769008
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CERTIFICATE

This is to certify that the thesis entitled "**Investigation into spontaneous combustion characteristics of some Indian coals and correlation study with their intrinsic properties**" submitted by Sri Shiva Shankar Jena (Roll No. 107MN010) in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in this thesis has not formed the basis for the award of any Degree or Diploma or similar title of any University or Institution.

Date:

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ABSTRACT

Mine fires and spontaneous combustion are prime issues in the coal mining industry and have led to huge coal losses and mine disasters. The property of self-oxidation of coal makes it highly vulnerable to degradation after being exposed to favorable conditions of spontaneous heating. For safe storage and use of coal, it is very important to know about its liability to spontaneous heating. It is known that, spontaneous heating is dependent upon intrinsic and extrinsic parameters. In the present work, the spontaneous combustion susceptibility of a number of coal samples belonging to different coalfields have been studies and its correlation with different intrinsic properties have been carried out.

In order to carry out the above study, 23 coal samples were collected from different Indian coalfields. Coal samples were collected from Singareni Collieries Company Ltd. (SCCL), Northern Coalfields Ltd.(NCL), Mahanadi Coalfields Ltd. (MCL), South Eastern Coal Fields Ltd. (SECL) and Eastern Coal Fields Ltd. (ECL).. The experiments carried out to determine the intrinsic properties and susceptibility indices were Proximate analysis, Bomb calorimetry, Crossing point temperature, Differential thermal analysis and Wet oxidation potential. The results of intrinsic properties are as presented in Table 1.

Table1: Intrinsic properties of the coal samples

Sl.No.	Sample Name	Proximate Analysis				Gross Calorific Values, GCV(Kcal/kg)
		M(%)	VM(%)	A(%)	FC(%)	
1	SCCL-1	4.15	38.27	13.6	43.98	6054.96
2	SCCL-2	4.39	24.71	33.19	37.71	5780.95
3	SCCL-3	6.08	27.16	17.63	49.13	5693.66
4	SCCL-4	4.34	37.52	13.67	44.47	4406.24
5	SCCL-5	4.38	34.87	23.34	37.41	5804.12
6	SCCL-6	4.38	38.39	9.97	47.26	6146.12
7	SCCL-7	5.1	32.23	12.59	50.08	5760.1
8	SCCL-8	6.76	18.32	36.58	38.34	3876.75
9	NCL-1	8	39.33	8	44.67	5710.57
10	MCL-1	2	30.75	30.24	37.01	5756.35
11	MCL-2	4.33	39.84	12.57	43.26	6163.01
12	MCL-3	6.39	33.29	25.35	34.97	4524.19
13	MCL-4	5.15	31.87	25.82	37.16	3948.47
14	MCL-5	7.65	31.91	13.35	47.09	6351.89
15	MCL-6	4.38	26.15	46.6	22.87	4377.9
16	MCL-7	6.03	28.92	30.6	34.45	5767.72
17	SECL-1	9.7	27.52	32.5	30.28	2366.5
18	SECL-2	12.05	29.26	26.42	32.27	4334.4
19	SECL-3	9.83	26.91	36.17	27.09	3884.86
20	SECL-4	5.57	13.7	61.34	19.39	2073.66
21	SECL-5	4.15	23.5	35.5	36.85	3190.79
22	SECL-6	8.2	24.32	34.61	32.87	3524.83
23	ECL-1	5.84	30.25	23.47	40.44	6092.22

Table 2: Susceptibility indices of the coal samples

Sl. No.	Sample Name	CPT (°C)	DTA				Wet Oxidation Potential Difference (mV)
			Critical Temp., T_c (°C)	Average slopes of stage			
				II A	II B	II	
1	SCCL-1	157	164.340	0.503	0.946	0.664	94.7
2	SCCL-2	178	130.490	0.421	0.313	0.363	127.6
3	SCCL-3	162	138.240	0.616	0.629	0.623	120.5
4	SCCL-4	159	163.000	0.525	1.174	0.780	124.2
5	SCCL-5	167	163.620	0.460	0.932	0.624	85.8
6	SCCL-6	166	169.150	0.311	0.637	0.421	88.3
7	SCCL-7	171	164.640	0.346	0.676	0.460	101.7
8	SCCL-8	175	123.400	0.501	0.318	0.395	107
9	NCL-1	177	178.950	0.388	0.787	0.506	92.4
10	MCL-1	172	180.720	0.299	0.998	0.461	114.7
11	MCL-2	166	178.310	0.385	0.951	0.518	115
12	MCL-3	167	166.550	0.361	0.519	0.414	121
13	MCL-4	168	163.900	0.451	0.768	0.538	110.9
14	MCL-5	170	162.560	0.438	0.452	0.442	102.3
15	MCL-6	160	184.950	0.557	1.582	0.823	93
16	MCL-7	185	161.370	0.649	1.165	0.819	126
17	SECL-1	159	123.620	0.907	0.531	0.721	98.3
18	SECL-2	144	113.610	1.366	0.541	0.857	104
19	SECL-3	160	127.050	0.520	0.252	0.385	116.8
20	SECL-4	168	342.310	0.007	4.563	0.472	110.3
21	SECL-5	172	167.250	0.090	0.241	0.135	122.4
22	SECL-6	162	133.750	0.504	0.328	0.420	115.3
23	ECL-1	168	157.510	0.498	1.023	0.685	93

From correlation analysis, it was found that average slopes of stage IIA and IIB obtained from DTA thermograms indicated a fairly accurate measure of spontaneous heating susceptibility as they showed high correlation with the intrinsic properties. Moisture and volatile matter content may also be helpful in assessment of liability of coal to spontaneous heating as they showed better correlation than other intrinsic properties viz., ash content and gross calorific values. Differential thermal analysis was found to give better correlation and so may be accepted as a better method for assessment of spontaneous heating susceptibility of coal than crossing point temperature and wet oxidation potential analysis. It is expected that the finding of this work will help the field engineers to accurately assess the spontaneous heating susceptibility of coal seams in their mines and plan the production activities accordingly.

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CHAPTER 1

INTRODUCTION

CHAPTER 1

1. INTRODUCTION

1.1 GENERAL

Spontaneous combustion as an unwelcomed feature had accompanied mining right from the days mining started up to the present day. There are several instances of fires caused due to spontaneous combustion in the history of coal mining which continued to burn for several decades and centuries. For instance, in India, the Jharia Fire consisting of several fires has continued to burn since 1923. The main problems that arise due to spontaneous combustion are related to the storage of coal as it leads to degradation of coal and possibilities of mine fires due to the process. Some of the other problems associated with spontaneous heating are degradation in heating values and coking properties, and in some cases serious environmental pollution. A careful investigation and assessment of these occurrences of fires reveals that most of these fires could have been averted if suitable preventive measures had been taken. So, spontaneous combustion of coal has attracted many researchers in the past and a variety of methods have been applied to understand the process and prevent it.

A number of approaches have been developed over the years in different countries to assess the proneness of a coal seam to spontaneous heating. This propensity to self-heating of coal also decides its incubation period, which decides the size of the panel to be formed, which is one of the most important design parameters in mine planning. It is therefore imperative that the planners of a mine determine in advance the spontaneous heating susceptibility of the coal seams to be mined so that either the coal has been extracted before the incubation period, or advance precautionary measures are planned to minimize the occurrences of spontaneous combustion.

Different methods have been followed in different coal producing countries to assess the spontaneous heating tendency of coal by different experimental techniques, viz. Crossing Point Temperature (CPT) in India, Russian U-index in Russia, Olpinski index in Poland, Adiabatic Calorimetry in U.S.A. etc. Some of the other methods attempted by researchers are Differential Thermal Analysis (Banerjee and Chakravarty, 1967; Gouws and Wade, 1989), Wet Oxidation Potential analysis (Singh et al., 1985; Tarafdar and Guha, 1989), Gas Indices studies (Panigrahi and Bhattacharjee, 2004; Singh et al., 2007), Critical Air

Blast (CAB) rate (Panigrahi and Sahu, 2005) etc.. However, there is no unanimity among researchers for the adoption of a particular method for the assessment of spontaneous heating susceptibility of coals, some of the researchers have suggested that a number of methods may be attempted to determine fairly accurately the degree of proneness of a particular coal to spontaneous heating.

1.2 OBJECTIVES

In order to determine the spontaneous combustion susceptibility of coal seams and to carry out correlation studies with their intrinsic properties, the current work has been planned with the following objectives:

- Collection of coal samples from mines of different Indian coalfields.
- Determination of intrinsic properties of the collected coal samples by proximate analysis and bomb calorimetry.
- Determination of spontaneous heating susceptibility by different experiments viz. crossing point temperature, wet oxidation potential analysis and differential thermal analysis.
- Correlation study to determine the extent of correlation between various intrinsic properties and susceptibility indices.

CHAPTER 2

LITERATURE REVIEW

CHAPTER 2

2.0 LITERATURE REVIEW

A lot of studies and research works have been carried out by different researchers in the past for assessment of spontaneous heating susceptibility of coal. Some of these works are summarized below:

Banerjee and Chakravarty (1967) suggested Differential Thermal Analysis (DTA) for the study of spontaneous combustion of coal, particularly in classifying coals with respects to their susceptibility to self-heating. They prescribed a standard experimental procedure for carrying out DTA experiments of coal where calcined alumina was recommended as inert reference material. A heating rate of 5°C/min was advised. The transition temperatures obtained from various coals were used to explain the phenomenon of self-heating of coal.

Banerjee (1972) determined the Crossing Point Temperatures (CPT) of different Indian coal samples by the Crossing Point Temperature method. He observed that coals with crossing point temperatures between 120°C & 140°C could be considered to be highly susceptible to spontaneous heating and those above 160°C, poorly susceptible. The moderately susceptible ones showed values in between the above mentioned.

Nandy et al. (1972) noted the variation in Crossing Point Temperature values with the moisture content, volatile matter, and the oxygen percentage of coal. He found that CPT decreased with the increase in each of these constituents. But beyond 35% V.M, 9% oxygen, or 4 to 6% moisture content there wasn't much variation in CPT values. In fact, above 4 to 6% moisture content (on as received basis), the CPT values showed a rising trend.

Feng et al. (1973) developed a composite liability index by using the results of Crossing Point Temperature experiments, called FCC index. This was calculated using the following equation:

$$\text{Liability Index} = \frac{\text{Average heating rate between 110°C and 220°C}}{\text{Relative ignition temperature}} \times 100$$

They selected 110°C as the lower limit in order to ensure that all the moisture had evaporated from the sample. The upper limit of 220°C was for the heating rate chosen as there would have been negligible release of volatile matter below this temperature.

Banerjee (1985) categorized different experimental techniques into three main groups: determination of chemical constituents of coal (rank correlation between moisture, volatile matter, ash content and oxygen percentage), thermal studies (CPT, DTA, DSC, H_2O_2, Puff temperature, Olpinski Index and Adiabatic Calorimetry) and Oxygen avidity studies (Peroxy complex Analysis, Russian U-Index and Wet oxidation).

Ghosh (1986) made an attempt to devise a method to identify coal's proneness to spontaneous combustion. They suggested that if pyrite is present in a coal in finely divided form, the proneness of coals towards spontaneous combustion increases; and that the temperature of a coal bed increases if water is added to it, which tends to indicate that water spraying or even flooding cannot be considered as an effective measure to control spontaneous combustion. Moreover, it was also suggested that if a coal body is chilled (to approximately -193 °C) the micropores and microcracks in the coal are possibly contracted. They suggested that atmospheric oxygen is less likely to enter the coal through micropores and microcracks; and hence chances of spontaneous combustion due to auto-oxidation are diminished.

Tarafdar and Guha (1989) carried out a preliminary investigation using wet oxidation method and they suggested that a systematic and thorough study is required to assess the significance of this technique.

Chandra etal. (1990) carried out a preliminary survey of the frequency of occurrence of fire due to spontaneous combustion in the different seams of the Raniganj Coalfield and showed the possibility of a relationship between coalification and spontaneous combustion of coals. Besides rank, as observed from reflectance studies, the amount of vitrinite and exinite contents of the coal seams also influenced the spontaneous heating tendency of the coal seams. They found that pyrite content of coal in the Raniganj Coalfields had no influence on the combustibility of the coal seams. It was concluded that the proneness to spontaneous heating of the coals is related to coalification. As the coalification increases, the susceptibility towards spontaneous combustion decreases gradually.

Bhatt and Agarwal (1995) devised a reaction-diffusion approach to consider the effects of moisture evaporation and condensation on the rate of oxidation of coal. They found that for a single isothermal particle, pseudo-steady-state balances on moisture and oxygen permit calculation of the effect of different levels of coal-bound moisture on the rate of oxidation. These species balances were combined with an energy balance to determine the particle temperature history during evaporation and condensation. It was shown that partial wetting of coal or condensation of moisture exerts two types of competing influences on the overall rate of oxidation. On one hand, a portion of the coal filled up with liquid moisture; in this region, the rate of oxidation became negligible, since the oxygen had to dissolve in the moisture before it can gain access to an active site on the coal surface and the solubility of oxygen in water was low. On the other hand, condensation also led to the release of the latent heat of vaporization. This heat effect raised the temperature of the particles and increased the rate of oxidation in the dry region of the coal. The relative magnitude of the rates of these competing influences determined whether the potential for spontaneous combustion was reduced or enhanced.

Pis et al. (1996) used differential thermal analysis (DTA) to study the self-heating behaviour of fresh and oxidized coals. Oxidation was performed in air at 200°C for periods of up to 72 hours. As the rank of the coal increased, both the self-heating and the end of combustion temperatures also increased. The total heat loss i.e. the area under the DTA curve increased with the rank of the coal. An increase in the self-heating temperature, a decrease in the temperature of the end of combustion and a decrease in total heat flow were observed after coal oxidation. A relationship was established between the total heat loss and the calorific value was determined using the ASTM standard.

Panigrahi et al. (1997) carried out experiments for the determination of Russian U-index. 10 coal samples from Jharia coalfields were analyzed using this method. The carbon, hydrogen, nitrogen and sulphur contents for these samples were determined using Fenton's method of ultimate analysis. The crossing point temperatures of these samples were also determined. Then, attempts were made to correlate the Russian index and CPT of coal samples with its constituents viz. carbon, hydrogen and ash contents. It was observed that from point of susceptibility towards spontaneous combustion,

Russian U-index shows similar relation with the basic constituents as shown by crossing point temperature, which may prove to be a handy method of coal categorization in Indian context.

Kuçuk et al. (2003)carried out an assessment of the spontaneous combustion characteristics of Askale lignite from Turkey. The effects of the gas flow rate, the moisture of the piles of coal, the humidity of the air and particle size on the spontaneous combustion characteristics of coal samples were analyzed using Crossing Point Method adapted to their laboratories conditions. The liability towards spontaneous combustion of this lignite was found to increase with decreasing particle size, increasing moisture content of the coal and decreasing humidity of the air.

Kadioglu et al. (2003) conducted an experimental study to evaluate the spontaneous combustion characteristics of two Turkish lignites moistened and air-dried at varying times. The contents of three predominant oxygen functional groups (carboxyl, hydroxyl, and carbonyl) of untreated, moistened and air-dried coal samples were also found outby wet chemical methods. It was found that the content of oxygen functional groups in moistened coal samples did not differ significantly from that of untreated coal samples. The liability towards spontaneous combustion of the two coals was found to reduce when moisture content increased with increase in contact time with water vapour.

Panigrahi et al. (2004) carried out extensive field studies to investigate the pillar fire problems in one coal mine in India. A Thermal IR gun and a Thermo vision camera had been employed for thermal scanning to assess the state of heating in selected pillars. Special sampling setups were designed to collect gas samples from the holes drilled into the selected pillars and the multi gas detector was used to analyze the composition of samples in situ. In order to predict the spontaneous heating in coal pillars, different gas ratios were calculated and it was observed that some of the established gas ratios, viz. Graham's ratio, Young's ratio etc. resulted in negative values in samples containing high amounts of methane. The modified gas ratios were proposed which may be useful for predicting the pillar fires. They suggested that these ratios could also be used for assessing the condition of fires in sealed-off areas.

Sahu et al. (2004) presented the method of finding out the spontaneous heating susceptibility of coal samples by using differential scanning calorimetry (DSC). 30 coal samples collected from 7 different Indian coalfields were studied by this method and the onset temperature for all the samples were determined. In addition, the crossing point temperatures (CPT) of all the samples were determined and a comparative study between onset temperature and CPT was also presented.

Sahu et al. (2005) described the determination of spontaneous heating susceptibility of coal samples by three different experimental methods viz., crossing point temperature (CPT), differential thermal analysis (DTA) and differential scanning calorimetry (DSC).They suggested that as all coals are not susceptible to spontaneous heating to the same extent, it was essential to assess their degree of proneness in order to plan advance precautionary measures. They also mentioned that the acceptability of a method for determining spontaneous heating characteristics of coal mainly depended upon how closely it predicted the spontaneous heating behavior in the field conditions. Considering this, it was concluded that the onset temperature obtained from differential scanning calorimetry may be a better method than crossing point temperature.

Singh et al. (2007) observed that in opencast mines, coal immediately oxidized and caught fire due to the intrinsic characteristics of coal, such as low rank, high moisture, high volatile matter, presence of sulfur in the form of pyrites, low crossing point temperature (CPT) and ignition point temperature (IPT) value and less incubation period. This paper presented different successful case studies regarding the safety management of open pit coal mines from occurrences of spontaneous heating.

Nelson etal. (2007) used a wide variety of techniques including oxidation mechanisms, ranking the propensity of different coals to self-heat, and the detection and suppression of self-heating to gain insight into the processes that govern the self-heating of coal. They found that moist coal in coal mines and stockpiles had very different combustion characteristics than those predicted on the basis of dry testing and therefore methods for ranking the propensity of coal to spontaneously combust in actual mining conditions needed to be developed.

Ahmed et al. (2008) calculated Liability index using CPT for studying the propensity of coal towards spontaneous heating. CPT that has been in lab requires extra precaution

for repeatable results. To overcome the difficulties, attempts were made to study the relationship between peripheral oxygen groups or the functional oxygen groups in coals and their correlation with proneness to auto-oxidation using liability index (LI).Also the correlation between liability index and crossing point temperature was presented in this paper.

Sahay et al. (2008) proposed realistic characterization of coal towards spontaneous heating for taking corrective measures. They developed a methodology based on thermo-decompositional study of coal sample for determination of minimum temperature at which coal bed temperature starts self-propellant known as critical temperature and a new liability index based on it. They presented a brief description of different liability index particularly based on thermal study, a critical analysis of dependency of critical temperature on moisture content, ash content, volatile matter, carbon content, surface area and porosity, new liability index based on thermo-decompositional study of coal sample and correlation with liability index model based on coal proximate analysis results of coal sample including surface area and porosity.

Beamish and Arisoy (2009)studied the effect of intrinsic properties on self-heating rates by adiabatic testing of a wide range of coals from Australia, New Zealand and Indonesia. They found that the relationship with coal rank was non-linear and could be expressed in the form of a third order polynomial for coals ranging in rank from sub-bituminous coals to low volatile bituminous coals. They recommended a coal ply sampling strategy to obtain site specific data regarding the effect of mineral matter on coal self-heating rates. They also found that coking coals had lower self-heating rates that steaming coals due to their high percentage of vitrinite.

Mohalik et al. (2009) presented the review of application of three thermal techniques viz. differential thermal analysis (DTA), thermogravimetry (TG) and differential scanning calorimetry (DSC); for studying the susceptibility of coal to spontaneous heating and fire. It also critically analyzed the experimental standards adopted by different researchers, while applying these techniques in studying thermal behavior of coal samples. The paper also presented the future direction of research in this subject area.

CHAPTER 3

THEORIES AND MECHANISM OF SPONTANEOUS HEATING

CHAPTER 3

3. THEORIES AND MECHANISM OF SPONTANEOUS HEATING

Spontaneous combustion of coal or other carbonaceous matter may be defined as the phenomenon of self-heating resulting eventually in its ignition without the application of external heat. The term is used generally for coal as spontaneous combustion of other minerals is very rare.

3.1 THEORIES OF SPONTANEOUS HEATING

The interaction of oxygen with coal, like all oxidation reactions, is exothermic. The heat from oxidation normally varies from 2.0 to 4.0 Cal/ml of O_2sorbed at N.T.P. Of course, there are other factors that might assist in the generation of heat, viz.

1. oxidation of pyrites present in coal,
2. bacterial action,
3. crushing of coal due to earth's movements, and
4. sorption of water vapour in coal.

But it is the oxidation of carbonaceous matter in coal that is primarily responsible for the initiation of spontaneous combustion.

3.1.1Pyrites theory

Heating due to oxidation of pyrites has been known to be a common phenomenon in pyrite mines. Coal contains pyrites, mainly in the form of iron pyrite, with concentrations at times exceeding 12 %. It was suspected earlier that heat evolved from aerial oxidation of pyrites is the cause of spontaneous heating of coal. However, it has now been established that pyrite present in coal might assist the oxidation of its carbonaceous content by breaking down coal into smaller fragments and exposing larger surface area to the air, as well as by elevating the temperature due to heat liberated from its own oxidation.

Winmill (1915-16) made thorough investigations on the influence of pyrites in the heating of coal in air. He concluded that pyrite can be the cause of heating only when it is present in considerable proportions and in very finely divided state. If the pyrite

occurs in lumps, or veins, or in the form of a highly crystalline 'brass', it contributes very little to the initial heating.

Graham (1923-24) corroborated Winmill's conclusions, and observed that when pyrite is present in finely divided state, it would rapidly absorb oxygen, He, however, concluded that most of the underground heating are due to oxidation of iron pyrite in moist air and is broadly represented by the following equation-

$$2\ FeS + 7\ 0_2 + 16\ H_2O \longrightarrow 2H_2S0_4 + 2\ FeSO_4.7H_20 + 316\ kcal$$

The above reaction shows an exothermic effect (316 kcal, i.e. 4.0 cal/ml of 0_2 consumed), It also yields reaction product having greater volume than the original pyrite, with the result that it would break open any coal in which they are embedded and thus exposing a greater surface of coal to the air.

Munzner (1975) inferred that pyrite might have an appreciable effect if its concentration in finely dispersed form exceeds 5 to 10%, if the pyrite present is less than 5 its effect can be neglected.

3.1.2Bacterial action

The contribution of heating due to the action of bacteria cannot be completely ruled out. In fact, spontaneous heating observed in haystacks and in wood are known to be mainly due to bacterial action.

Potter(1908)reported some heating from bacterial action in coal, inoculating with a pure culture of diplococcus.

Galle, Winmill, Graham, Fuchs and others also investigated the possibility of heating in coal due to bacterial reaction.

Fuchs (1927), however, concluded that bacteria could cause only a slight heating which may not play any significant role.

2.1.3 Heating due to earth movements

The crushing of coal under the pressure of superincumbent strata may at times be a source of heating contributory to spontaneous combustion. Sudden roof fall and the resultant adiabatic compression of air may also elevate the ambient temperature temporarily. Briggs observed that though the heat from crushing could contribute to spontaneous combustion, the oxidation of coal is more important a factor. Crushing of

coal, of course, increases the exposed surface of coal for oxidation, making it more potent for self-heating.

2.1.4Sorption of water in coal

When a liquid penetrates into coal (partly in vapour form), the coal starts swelling up, requiring some amount of energy. But more energy is liberated by the interaction between the fluid and the coal surface, and is known as heat of wetting. Coal being hygroscopic in nature will generate large amount of heat when wetted with water. Bhattacharya observed that besides the above mentioned heat caused due to wetting, when water is absorbed as vapor, its latent heat of vaporization is added to the heat of wetting and thereby increases the coal bed temperature considerably.

3.2 MECHANISM OF SPONTANEOUS COMBUSTION

When coal is exposed to air it absorbs oxygen at the Imposed surface. Some fractions of the exposed coal substance absorb oxygen at a faster rate than others and the oxidation results m formation of gases, mainly CO, CO_2 and water vapor along with evolution of heat during the chemical reaction. The process takes place eyen at normal atmospheric temperature but it is slow and the heat evolved is not perceptible as it is carried away by the air unless the latter is stagnant. If, however, the rate of dissipation of heat is slow compared with the evolution of heat by oxidation, there is a gradual build-up of heat and slow rise in- the temperature of coal. At the raised temperature the process of oxidation is slightly accelerated and some other fractions of coal become susceptible to oxidation. A stage is reached when the build-up of heal and the rise of tempera tare reaches the ignition point of coal which then catches fire. A good air current will effective1y prevent undue increase of temperature; absence of air will prevent oxidation; and somewhere between these two extremes conditions may permit marked heating to take place Once the coal reaches its ignition point (as distinct from slow oxidation), the air supply to it will only increase the combustion. The ignition temperature of bituminous coal is nearly 200°C and of anthracite coal, nearly 398°C. The coal may be smoldering in the beginning but it may soon break up into flames if sufficient oxygen or fresh air feeds the hot coal. This process of self-heating of coal resulting ultimately in its combustion is known as spontaneous combustion. The different stages in which this process continues is shown below:

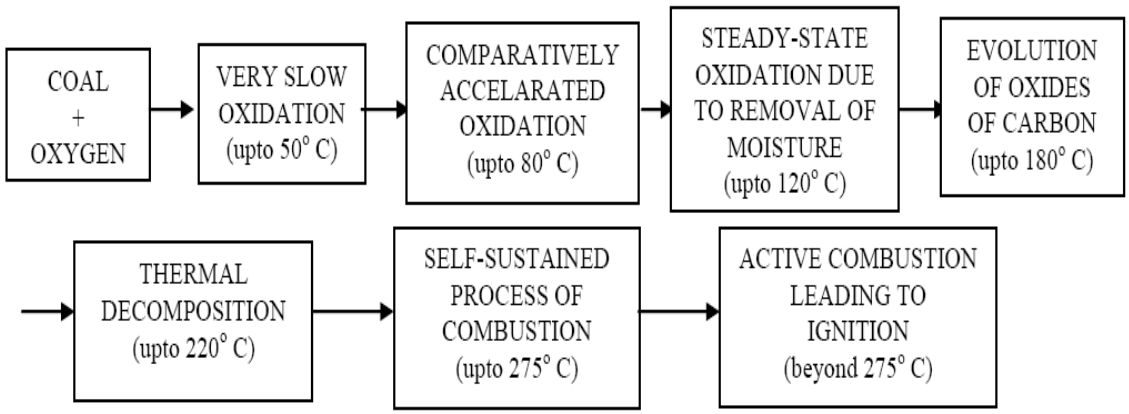


Figure 2.1: Stages in spontaneous heating of coal

3.3 FACTORS AFFECTING SPONTANEOUS HEATING OF COAL

Spontaneous heating occurs only in certain seams and these susceptible seams are frequently adjacent to seams worked by the same method and subjected to the same tectonics, but which have never been the site of combustion. A few seams have only been susceptible in one area and, again, some seams are more susceptible than others. In this chapter the effect of all the factors on spontaneous heating susceptibility of coals are discussed.

Every incident of spontaneous combustion, however small, if not dealt with effectively and efficiently in the early stages can develop into open fires or into an explosion of gas or dust, with devastating results.

The whole phenomenon of the spontaneous combustion is an aggregate effect of the three factors viz., seam factors, geological factors and mining factors.

A) **Seam factors**

- Rank
- Petrographic Composition
- Temperature
- Available Air
- Particle Size
- Moisture
- Sulphur
- Effect of Previous Oxidation
- Physical Properties
- Heating due do Earth Movement
- Bacteria
- Other Minerals

B) Geological factors

- Seam thickness
- Seam gradient
- Caving characteristics
- Faulting
- Coal outbursts
- Friability
- Depth of cover

C) Mining Factors

- Mining methods
- Rate of advance
- Pillar conditions
- Roof condition
- Crushing
- Packing
- Effect of timber
- Leakage
- Multi-seam working
- Coal losses
- Main roads
- Worked out areas
- Heat from machines
- Stowing
- Ventilating pressure
- Change in humidity

CHAPTER 4

EXPERIMENTAL INVESTIGATIONS

CHAPTER 4

4. EXPERIMENTAL INVESTIGATIONS

In this study, a total of 23 samples were collected from different mines of Singareni Collieries Company Ltd. (SCCL), Northern Coalfields Ltd. (NCL), Mahanadi Coalfields Ltd. (MCL), South Eastern Coalfields Ltd. (SECL) and Eastern Coalfields Ltd. (ECL) were collected by channel sampling method. The intrinsic properties of the coal samples were determined by proximate analysis and bomb calorimetry. The spontaneous heating susceptibility indices were found out by Crossing Point Temperature (CPT), Differential Thermal Analysis (DTA) and Wet Oxidation Potential Difference (WOPD) methods.

4.1 SAMPLE COLLECTION AND PREPARATION

Sampling is a process by which very small portions of an ore body are collected and analyzed to estimate the average mineral content of the ore body. Sampling of an ore body is carried out for the first time in the life of the mine during the stage of prospecting. Trenches cut at the time of prospecting, the cuttings of churn drilling and the cores of diamond drilling offer valuable samples to the prospector. Their analysis result decides the future course of prospecting.

In the development stage samples are collected from the roadways driven in the ore body, i.e. from the levels or drives, raises and winzes. Rarely are samples collected from crosscuts unless mineralization is observed to extend to the cross-cuts also, where the drives are made in the footwall, the ore body is sampled by means of crosscut raises at regular intervals. At the time of stopping such crosscut raises serve as ore chutes for removal of broken ore.

Four routine sampling methods are suitable for specific objectives in the daily mine routine.

1. Channel or groove sampling.
2. Chip sampling.
3. Grab sampling.
4. Bulk sampling.

4.1.1Sample collection by channel sampling

In this project, the samples were collected by channel sampling method as described in IS 436 Part I/Section I - 1964. The section of seam to be sampled was exposed from the roof to the floor. The seam sample was taken in a channel representing the entire cross-section of the seam having the dimensions of 30 x 10 cm, that is, 30 cm in width and 10 cm in depth.

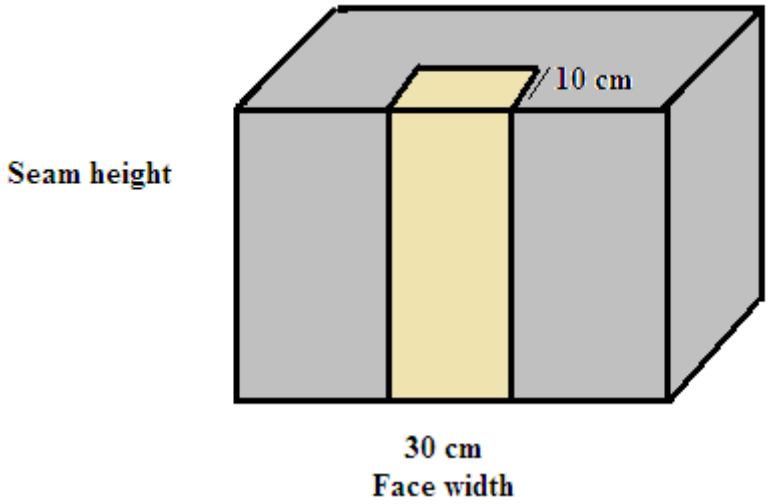


Figure 4.1: Channel sampling

For this purpose, two parallel lines, 30 cm apart end at right angles to the bedding planes of the seam were marked by a chalked string on the smooth, freshly exposed surface of the seam. Obvious dirt bands exceeding 10 cm in thickness were excluded. The channel between the marked chalk lines in the seam was cut to a depth of 10 cm and the coal sample was collected on a clean strong cloth or tarpaulin placed immediately at the bottom so that the chances of pieces flying off during excavation of coal were minimized.

4.1.2 Sample preparation

The samples collected from the coalfields by channel sampling were brought to the laboratory and crushed as per the experimental requirements. To get a representative sample of the entire coal sample, coning and quartering procedure was done. The crushed sample is then sieved to -212 µ size and stored in air tight polythene packets. The packets were stored in air tight containers for further use in experimentation.

All 23 samples were collected and prepared by following the above procedures. 8 samples belonged to SCCL, 1 from NCL, 7 from MCL, 6 from SECL and 1 from ECL. The samples were first numbered as shown below and then analyzed.

Table 4.1: Samples collected for the investigation

Sl. No.	Sample Name
1	SCCL-1
2	SCCL-2
3	SCCL-3
4	SCCL-4
5	SCCL-5
6	SCCL-6
7	SCCL-7
8	SCCL-8
9	NCL-1
10	MCL-1
11	MCL-2
12	MCL-3
13	MCL-4
14	MCL-5
15	MCL-6
16	MCL-7
17	SECL-1
18	SECL-2
19	SECL-3
20	SECL-4
21	SECL-5
22	SECL-6
23	ECL-1

4.2DETERMINATION OF INTRINSIC PROPERTIES OF COAL

4.2.1 Proximate analysis (IS 1350 part I -1984)

Proximate analysis of the coal samples was carried out as per procedures mentioned in IS 1350 - part I (1984) to determine the amount of moisture content (M), volatile matter (VM), ash content (A) and fixed carbon (FC) in the coal samples. The variables are then converted to different basis as mentioned below:

- air-dried: It neglects the presence of moistures other than inherent moisture.
- dry-basis: It leaves out all moistures, including surface moisture, inherent moisture, and other moistures.
- dry ash free basis (daf): It neglects all moisture and ash constituent in coal.
- dry mineral matter free basis (dmf): It leaves out the presence of moisture and mineral matters in coal.

Calculation on different bases was done by multiplying the following:

- Dry basis: (100 / 100 – M)
- Dry ash free basis: (100 / 100 – (M + A))
- Dry mineral matter free basis: [100 / (100 – (M + MM)]

Where, M: Moisture content

V: Volatile matter content

A: Ash content

MM: Mineral Matter = 1.1 * A

4.2.1.1 Determination of Moisture Content

Coal is always associated with some amount of moisture, which is both physically and chemically bound, due to its nature, origin and occurrence. When a wet coal is exposed to atmosphere, the external moisture evaporates. The apparently dry coal still contains some moisture, which can only be removed on heating beyond 100°C. Inherent moisture is termed as equilibrium or air-dried or hygroscopic moisture as it is related to the inherent hygroscopic nature of the coal whereas external moisture is called accidental or free moisture as it depends mainly on the mode of occurrence and handling of coal.

In this work, moisture content of the coal samples was found out by taking about 1g of finely powdered (-212 μ) air-dried coal sample in a watch glass and then placing it inside a moisture oven (Figure 4.2) maintained at 108±2 °C. It was allowed to remain in

the oven for 1.5 hours and was then taken out with a pair of heat resistant gloves, cooled in a desiccator for about 15 minutes and then weighed. The loss in weight was reported as moisture (on percentage basis).

The calculation was done as per the following.

Total moisture content of the original sample,

$$\% \text{ Moisture} = \frac{Y - Z}{Y - X} \times 100$$

Where, X = weight of empty crucible, g

Y = weight of crucible + coal sample before heating, g

Z = weight of crucible + coal sample after heating, g

Y - X = weight of coal sample, g

Y- Z = weight of moisture, g

4.2.1.2 Determination of Volatile matter

When coal sample is heated in a closed silica crucible kept in muffle furnace under anaerobic conditions, the loss of mass corrected for moisture is referred to as volatile matter. The matter lost is the product of thermal decomposition of the various components of coal. Some of the constituents of coal volatile matter are hydrogen, carbon monoxide, methane, other hydrocarbons, tar vapours, ammonia, some organic sulphur, oxygen containing compounds and some incombustible gases, such as carbon dioxide and water vapour.

In this work, a special volatile matter silica crucible (38mm height, 25mm external diameter and 22mm internal diameter) was used. The empty volatile matter crucible was weighed. Approximately 1g of coal sample of -212µ (BSS)size was weighed in the volatile matter crucible and it was placed inside a muffle furnace (Fig. 4.3) maintained at 925±5 °C with the lid covering the crucible. The heating was carried the muffle furnace out exactly for seven minutes, after which the crucible was removed, cooled in air, then in a desiccator and weighed again. The calculation was done as per the following.

$$\% \text{ Volatile matter} = \frac{Y - Z}{Y - x} \times 100 - M$$

Where X = weight of empty crucible, g

Y = weight of crucible + coal sample before heating, g

Z = weight of crucible + coal sample after heating, g

Y-X = weight of coal sample, g

Y- Z = weight of volatile matter + moisture, g

4.2.1.3 Determination of Ash content

Coal ash is the residue remaining after the combustion of coal under specified conditions. It does not occur as such in the coal, but is formed as the result of chemical changes that take place in the mineral matter during the combustion process. Ash and mineral matter of coal are therefore not identical.

There are two types of ash forming materials in coal: extraneous and inherent mineral matters. The extraneous mineral matter consists of materials such as calcium, magnesium and ferrous carbonates, pyrite, marcasite, clays, shales, sand and gypsum. The extraneous mineral matter owes its origin to

i) the substances which got associated with the decaying vegetable material during its conversion to coal, which is difficult to remove by mechanical methods, and

ii) rocks and dirt getting mixed up during mining and handling of coal. Inherent mineral matter represents the inorganic elements combined with organic components of coal. The origin of such materials is probably the plant materials from which the coal was formed.

Ash from inherent mineral matter is insignificant as far as the total quantity of ash is concerned. But Indian coals suffer from the major disadvantage, that the mineral matter content is not only high, but of intimately associated type, due to its drift origin.

In this work, the procedure followed for ash content determination is described as follows. The empty silica crucible was cleaned by heating in a muffle furnace for one hour at 800°C so that other mineral matters if present get burnt. It was taken out, cooled to room temperature and the weight is taken. Approximately 1gm of coal sample of -212µ (BSS) size was weighed in the crucible and placed in a muffle furnace at 450°C for 30 minutes and the temperature of the furnace was raised to 850°C for 1 hour. The crucible was taken out and placed in a desiccator and weighed.

$$\% \text{ Ash} = \frac{Z - X}{Y - X} \times 100$$

Where X = weight of empty crucible in grams

Y = weight of coal sample + crucible in grams (Before heating)

Z = weight of coal sample + crucible in grams (After heating)

Y - X = weight of coal sample, g

Z - X = weight of ash, g

4.2.1.4 Determination of Fixed carbon

It is determined by subtracting the sum of all the above parameters (in air dried basis) and is given as

Fixed Carbon, FC = 100 – (M + V + A)

Where, M: Moisture content

V: Volatile matter content

A: Ash content

The results of proximate analysis are as presented in table 4.2.

Table 4.2: Results of proximate analysis

Sl. No.	Sample Name	Basis	Moisture (%)	Volatile Matter (%)	Ash (%)	Fixed Carbon (%)
1	SCCL-1	air dried	4.15	38.27	13.6	43.98
		dry	-	39.93	14.19	45.88
		daf	-	46.53	-	53.47
		dmf	-	45.63	-	54.37
2	SCCL-2	air dried	4.39	24.71	33.19	37.71
		dry	-	25.84	34.71	39.44
		daf	-	39.59	-	60.41
		dmf	-	36.19	-	63.81
3	SCCL-3	air dried	6.075	27.16	17.63	49.14
		dry	-	28.92	18.77	52.31
		daf	-	35.60	-	64.40
		dmf	-	34.08	-	65.92
4	SCCL-4	air dried	4.34	37.52	13.67	44.47
		dry	-	39.22	14.29	46.49
		daf	-	45.76	-	54.24
		dmf	-	44.84	-	55.16
5	SCCL-5	air dried	4.38	34.87	23.34	37.41
		dry	-	36.47	24.41	39.12
		daf	-	48.24	-	51.76
		dmf	-	46.52	-	53.48
6	SCCL-6	air dried	4.38	38.39	9.97	47.26
		dry	-	40.15	10.43	49.42
		daf	-	44.82	-	55.18
		dmf	-	44.17	-	55.83

Sl. No.	Sample No.	Basis	Moisture (%)	Volatile Matter (%)	Ash (%)	Fixed Carbon (%)
7	SCCL-7	air dried	5.1	32.23	12.59	50.08
		dry	-	33.96	13.27	52.77
		daf	-	39.16	-	60.84
		dmf	-	38.21	-	61.79
8	SCCL-8	air dried	6.76	18.32	36.58	38.34
		dry	-	19.65	39.23	41.12
		daf	-	32.33	-	67.67
		dmf	-	27.66	-	72.34
9	NCL-1	air dried	8	39.33	8	44.67
		dry	-	42.75	8.70	48.55
		daf	-	46.82	-	53.18
		dmf	-	46.31	-	53.69
10	MCL-1	air dried	2	30.75	30.24	37.01
		dry	-	31.38	30.86	37.77
		daf	-	45.38	-	54.62
		dmf	-	42.83	-	57.17
11	MCL-2	air dried	4.33	39.84	12.57	43.26
		dry	-	41.64	13.14	45.22
		daf	-	47.94	-	52.06
		dmf	-	47.14	-	52.86
12	MCL-3	air dried	6.39	33.29	25.35	34.97
		dry	-	35.56	27.08	37.36
		daf	-	48.77	-	51.23
		dmf	-	46.79	-	53.21
13	MCL-4	air dried	5.15	31.87	25.82	37.16
		dry	-	33.60	27.22	39.18
		daf	-	46.17	-	53.83
		dmf	-	44.08	-	55.92
14	MCL-5	air dried	7.65	31.91	13.35	47.09
		dry	-	34.55	14.46	50.99
		daf	-	40.39	-	59.61
		dmf	-	39.37	-	60.63

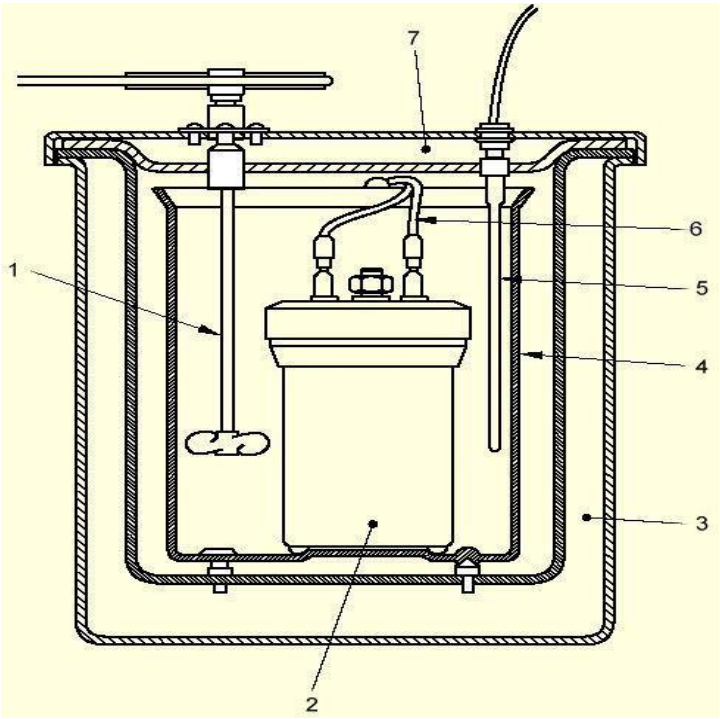
Sl. No.	Sample No.	Basis	Moisture (%)	Volatile Matter (%)	Ash (%)	Fixed Carbon (%)
15	MCL-6	air dried	4.38	26.145	46.6	22.88
		dry	-	27.34	48.73	23.92
		daf	-	53.34	-	46.66
		dmf	-	48.43	-	51.57
16	MCL-7	air dried	6.03	28.92	30.06	34.99
		dry	-	30.78	31.99	37.24
		daf	-	45.25	-	54.75
		dmf	-	42.55	-	57.45
17	SECL-1	air dried	9.7	27.52	32.5	30.28
		dry	-	30.48	35.99	33.53
		daf	-	47.61	-	52.39
		dmf	-	44.49	-	55.51
18	SECL-2	air dried	12.05	29.26	26.42	32.27
		dry	-	33.27	30.04	36.69
		daf	-	47.55	-	52.45
		dmf	-	45.20	-	54.80
19	SECL-3	air dried	9.83	26.91	36.17	27.09
		dry	-	29.84	40.11	30.04
		daf	-	49.83	-	50.17
		dmf	-	46.23	-	53.77
20	SECL-4	air dried	5.57	13.7	61.34	19.39
		dry	-	14.51	64.96	20.53
		daf	-	41.40	-	58.60
		dmf	-	28.07	-	71.93
21	SECL-5	air dried	4.15	23.5	35.5	36.85
		dry	-	24.52	37.04	38.45
		daf	-	38.94	-	61.06
		dmf	-	35.12	-	64.88
22	SECL-6	air dried	8.2	24.32	34.61	32.87
		dry	-	26.49	37.70	35.81
		daf	-	42.52	-	57.48
		dmf	-	38.82	-	61.18
23	ECL-1	air dried	5.84	30.25	23.47	40.44
		dry	-	32.13	24.93	42.95
		daf	-	42.79	-	57.21
		dmf	-	40.83	-	59.17

4.2.2 Determination of Gross Calorific Value by Bomb Calorimetry

The energy value of coal or calorific value is the amount of potential energy in coal that can be converted into actual heating ability. The value can be calculated and compared with different grades of coal or even other materials. Materials of different grades will produce differing amounts of heat for a given mass. The calorific value of coal is usually determined by the bomb calorimeter method.

4.2.2.1Bomb Calorimeter

Bomb calorimeter consists of a stout cylindrical chamber known as bomb (Fig. 4.2) of stainless steel. This chamber is fitted with an air tight cover which can be screwed on the chamber. The cover has three terminals; two for sparking and one for the entry of oxygen. After forcing the oxygen into the chamber the passage can be blocked by screwing in the third terminal. The Schematic layout of bomb calorimeter is shown below in Fig. 4.4.



1) Stirrer 2) Calorimeter bomb 3) Jacket 4) Calorimeter vessel
5) Platinum Resistance Thermometer (PRT) 6) Ignition lead7) Jacket

Figure 4.2: Schematic layout of Bomb Calorimeter

On the other side of the cover, there are two bent rods connected to two terminals. The bent rods have small holes through which two fuse wires are connected. There is the provision for putting the crucible containing the pellet tied to the fuse wire by means of

a cotton thread. This whole set-up is placed in a bigger vessel containing a known quantity of water in it. The vessel is jacketed to minimize the heat loss by radiation. A stirrer is used for stirring the water in the bigger vessel. There is a provision for inserting the thermometer.

4.2.2.2 Experimental Procedure

The calorific values of all the coal samples were determined following IS-1350, Part-II (2000). Approximately about 1gram of -212µ size air dried coal sample was taken by weighing in a balance. A pellet was made with the coal and weighed. The calorimeter cover was taken and about 10 cc of distilled water was poured into it. The pellet in the crucible was brought in contact with the fuse wire by means of a thread. The cover was then tightened. Oxygen was then admitted into the calorimeter at a pressure of about 25 atmospheres. 2 liters of water was put into the bigger vessel. The thermometer was inserted into the pocket. Necessary electrical connections were made and stirrer was adjusted in the corrected position. The stirring was done gently for five minutes. The initial temperature reading was then taken. The bomb was fired. Sparking and combustion of coal took place in the calorimeter. The maximum reached temperature was then noted. The bomb was removed and the pressure released. The bomb interior was examined for unburnt or sooty deposits. If such material was found, the test was discarded. The photograph of the experimental setup of bomb calorimeter is shown below in Fig. 4.3.

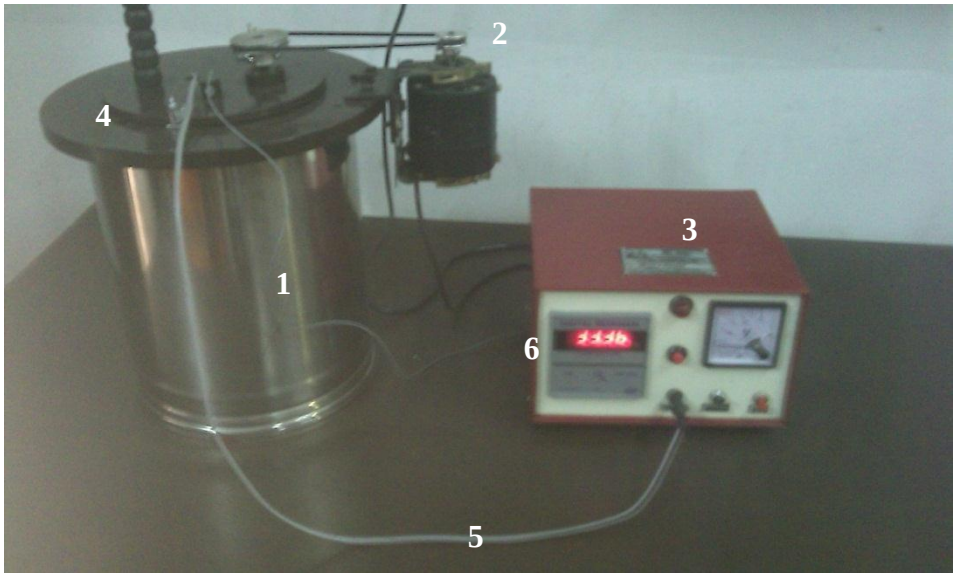
The GCV (Gross Calorific Value) was calculated as,

$$\mathrm{GCV} = \frac{2366.5 \times \Delta T}{m}$$

where ΔT : Difference between Initial and Final Temperatures and

m: mass of the pellet

The constant 2366.5 is the specific heat capacity of water in Kcal/°C



1. Calorimeter vessel 2. Stirrer 3. Bomb 4. Read out unit 5. Firing cable 6.Temperature reading

Figure 4.3: Photographic view of the experimental set-up of bomb calorimeter

The calorific values of all the coal samples determined by following the above procedure is as presented in table 4.3.

Table 4.3: Gross calorific values of the coal samples

Sl. No.	Sample Name	(GCV) (Kcal/kg)
1	SCCL-1	6054.96
2	SCCL-2	5780.95
3	SCCL-3	5693.66
4	SCCL-4	4377.9
5	SCCL-5	5804.12
6	SCCL-6	6146.12
7	SCCL-7	5760.1
8	SCCL-8	5767.72
9	NCL-1	5710.57
10	MCL-1	5756.35
11	MCL-2	6163.01
12	MCL-3	4524.19
13	MCL-4	3948.47

14	MCL-5	6351.89
15	MCL-6	3876.75
16	MCL-7	6092.22
17	SECL-1	2366.5
18	SECL-2	4334.4
19	SECL-3	3884.86
20	SECL-4	2073.66
21	SECL-5	3190.79
22	SECL-6	3524.83
23	ECL-1	4406.24

4.3 DETERMINATION OF SUSCEPTIBILITY INDICES

4.3.1 Crossing Point Temperature (CPT)

This method envisages heating of coal sample in an oxidizing atmosphere at a definite programmed rate of temperature rise. The lowest temperature at which the exothermic reactions in coal bed becomes self-propellant is termed as Crossing Point Temperature (CPT). In other words, crossing point temperature is the temperature at which the temperature of coal in the reaction tube coincides with the bath temperature. The photograph of the experimental setup is shown below in Fig. 4.4.

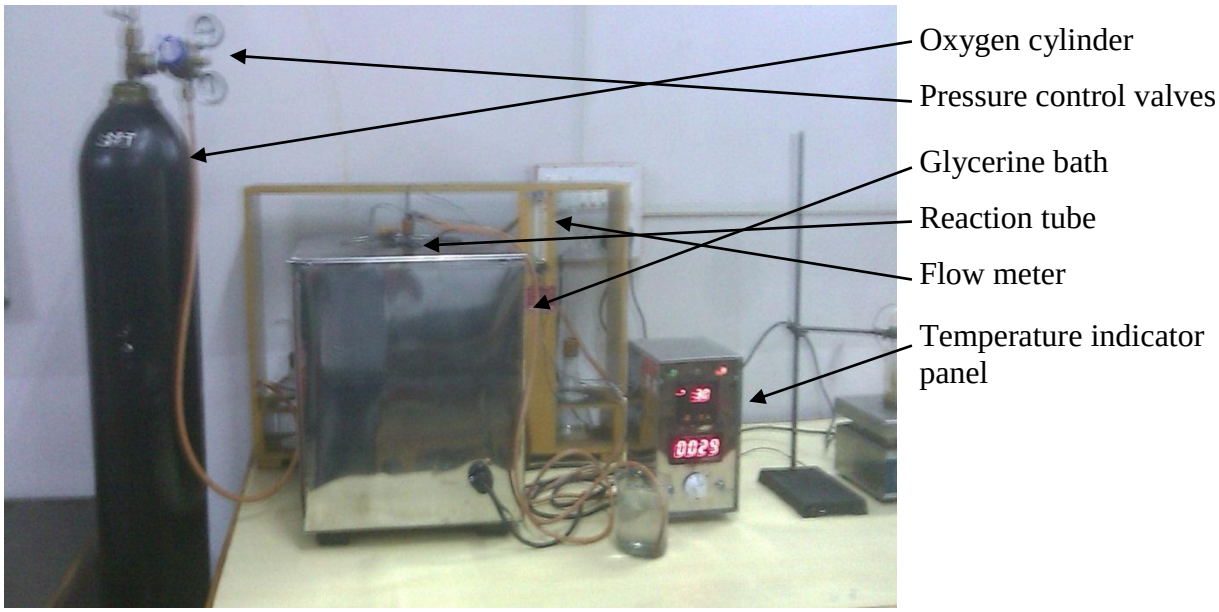


Figure 4.4: Photographic view of CPT Set-up

4.3.1.1 Experimental Procedure

In this work, 20gm sample of size -212µ was placed in the reaction tube with glass wool at the bottom most position. The tube was then lightly tapped a fixed number of times to achieve uniform packing density of the samples. The reaction tube was then placed in the furnace and a chromel-alumel thermocouple was inserted at the center of the sample. The furnace was switched on and simultaneously air was allowed to pass through the sample, with an average heating rate of 5°C per minute and air flow rate of 80ml/ min. The temperatures of the glycerine bath and the coal sample were recorded at every five minute interval till the temperature of coal crossed over and went beyond the bath temperature. Figure 4.5 is an example of the time-temperature plot obtained from CPT experiment of the coal sample SCCL-4.From the plot it can be inferred that initially due to release of moisture from coal sample the temperature of coal decreases but after that the sample temperature is found to be parallel to the bath temperature for some time. After that the temperature suddenly increases at a faster rate and crosses the bath temperature and rises steeply beyond that. The point on the graph at which the sample temperature crosses the bath temperature is called as Crossing Point Temperature (CPT). Lower the CPT, higher is the susceptibility of the coal sample to catch fire. The CPT of all the coal samples is as presented in table 4.4. The susceptibility of coal samples to spontaneous combustion (table 4.5) is assessed following the criteria specified by Mahadevan and Ramlu (1985).

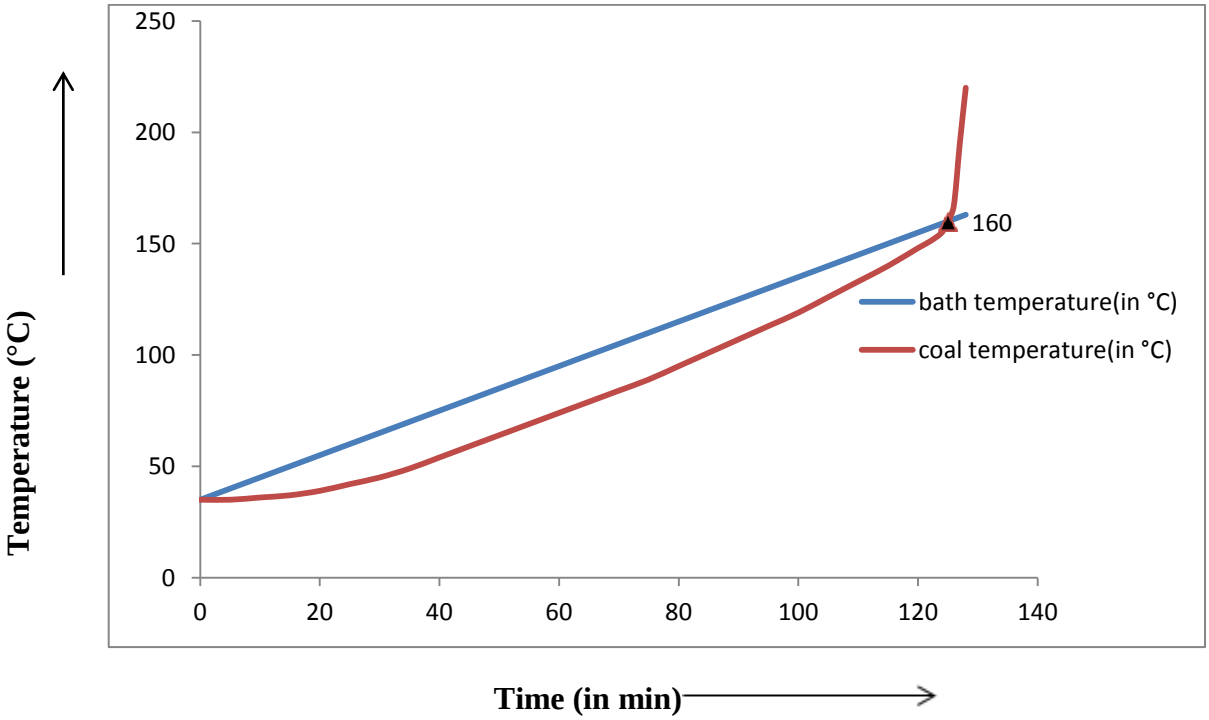


Figure 4.5: Determination of CPT for Sample SCCL-4

Table 4.4: Crossing point temperature of coal samples

Sl. No.	Sample Name	CPT (°C)
1	SCCL-1	157
2	SCCL-2	178
3	SCCL-3	162
4	SCCL-4	159
5	SCCL-5	167
6	SCCL-6	166
7	SCCL-7	171
8	SCCL-8	175
9	NCL-1	177
10	MCL-1	172
11	MCL-2	166
12	MCL-3	167
13	MCL-4	168
14	MCL-5	170
15	MCL-6	160
16	MCL-7	185
17	SECL-1	159
18	SECL-2	144
19	SECL-3	160
20	SECL-4	168
21	SECL-5	172
22	SECL-6	162
23	ECL-1	168

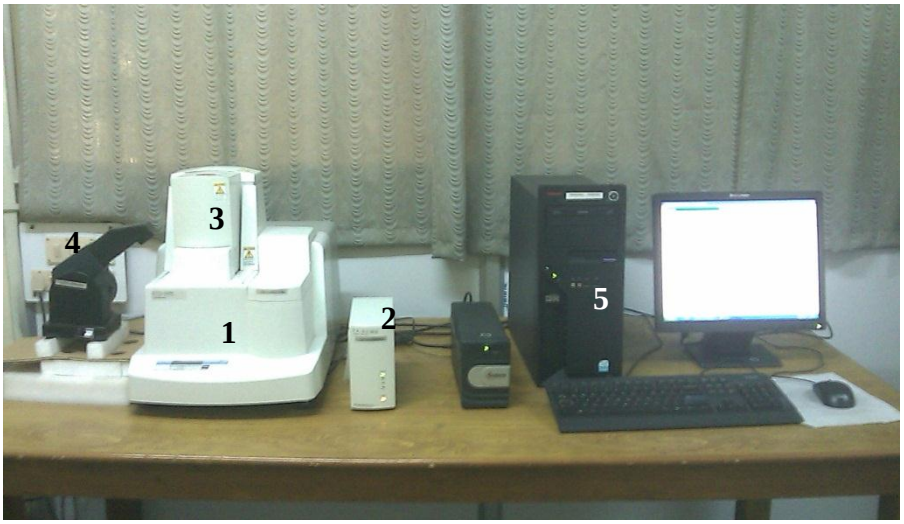
Table 4.5: Risk rating criteria based on CPT values

CPT (^{0}C)	Risk
120-140	Highly liable to self- heating
140-160	Moderately liable to self- heating
160-180	Less liable to self-heating

4.3.2 Differential Thermal Analysis (DTA)

Differential thermal analysis involves heating a small test specimen at a constant rate and continuously recording the instantaneous temperature difference (ΔT) between it and as identically heated inert reference material as a function of sample temperature (T). The resultant thermogram, a record of ΔT against T, with its characteristics heat changes and intensities, depicts the physical or chemical changes of the material at that particular temperature and is characteristic of that material used.

The DTA apparatus consists of a sample and reference holder, a furnace, a temperature programmer to maintain constant temperature rate and an output to monitor the test (Fig. 4.6). The sample holder has two thermocouples each for reference and sample. The sample is contained in a small crucible. The thermocouple should not be in direct contact with sample. By the temperature programmer the temperature is made to rise at a constant rate and the temperature difference of the sample and reference is plotted against time.



1) Differential Thermal Analyzer(DTA- 60H) 2) TA – 60 WS Collection Monitor
3) Tubular Furnace 4) Blower 5) Computer for Data Processing

Figure 4.6: Photograph of the experimental setup of DTA

In DTA the temperature difference between sample and inert reference is measured when both are subjected to identical heat treatments and then plotted against time or temperature.

4.3.2.1 Experimental Procedure

In this work, the DTA experiment was carried out by a thermal analyzer by following the standardized parameters suggested by Banerjee and Chakravarty (1967). The crucible for sample and reference was put in position on the ceramic post. A small amount of coal sample (-212µ) was put onto the sample holder. Oxidizing atmosphere was maintained by keeping the coal sample exposed to air. Alpha alumina powder was used as the reference material. The tubular furnace was then lowered. The software was programmed to run until 450^0C at a rate of 5^0C per minute. After the heating is terminated, a thermogram is obtained for the sample. DTA thermograms of all the coal samples have been presented in figures A1 to A23 in Appendix-I.

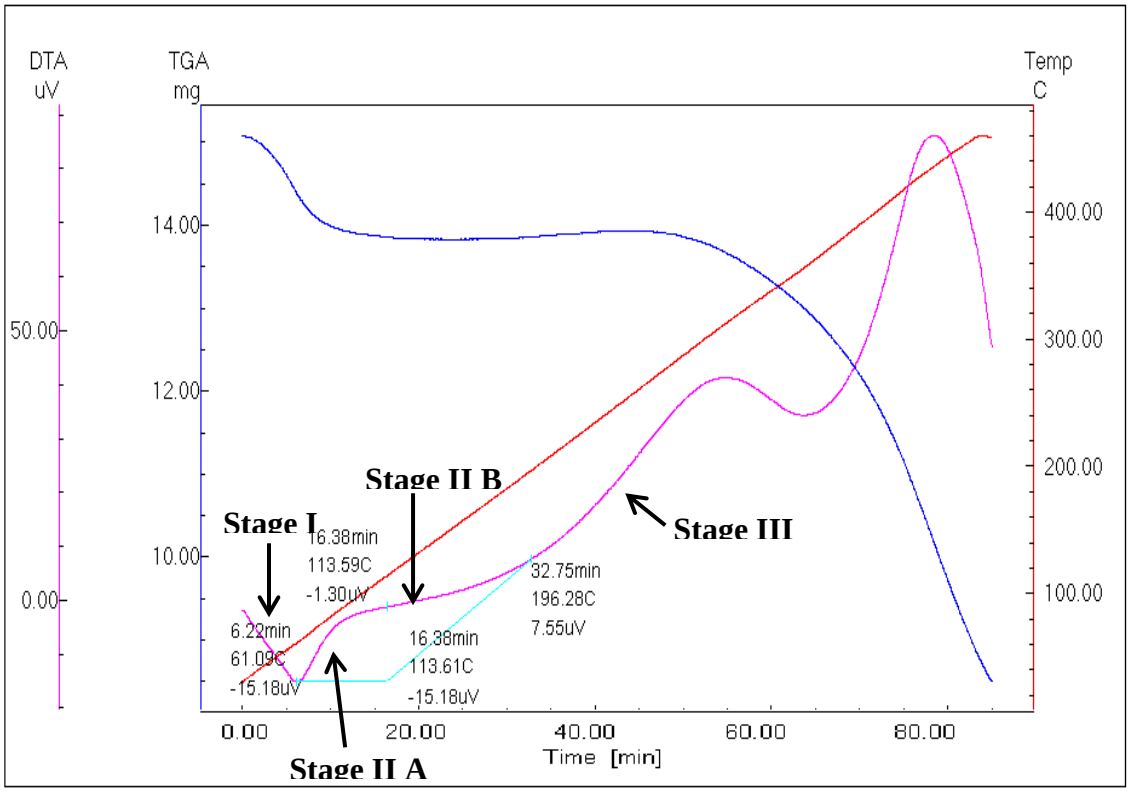


Figure 4.7: DTA thermogram for sample SECL-2

The plot consists of three parts: stage I, II, III as shown in figure 4.7, which shows the DTA curve obtained from coal sample SECL-2. During stage I, mostly endothermic reactions take place and the temperature falls due to release of moisture. Stage II consists of two parts, IIA and IIB. From the beginning of IIA the heating tendency starts accompanied by a small amount of endothermic reactions. The point where IIA begins is called as the inflexion point as the thermogram suddenly rises. In IIB

exothermic reactions start to dominate. The beginning of stage III is called as Transition temperature. This temperature is very important as it is directly related to spontaneous heating tendency of coal. The lower the transition temperature, higher is the susceptibility of coal to spontaneous combustion and vice versa. From stage III onwards complete exothermic reactions occur and the temperature continues to rise steeply. The slopes of the DTA thermogram are also taken as indicators of spontaneous combustion. Lower the slope values lower is the susceptibility. The transition temperature was found out by drawing tangents at the inflexion point and the highest slope stage III. Their intersection gave the transition temperature T_c. The slopes and the T_c were determined by using the software available with the equipment and have been presented in table 4.6.

Table 4.6: Transition temperatures and slope values obtained from DTA thermograms

Sl. No.	Sample Name	T_c(°C)	Slope II a	Slope II b	Slope II
1	SCCL-1	164.340	0.503	0.946	0.664
2	SCCL-2	130.490	0.421	0.313	0.363
3	SCCL-3	138.240	0.616	0.629	0.623
4	SCCL-4	163.000	0.525	1.174	0.780
5	SCCL-5	163.620	0.460	0.932	0.624
6	SCCL-6	169.150	0.311	0.637	0.421
7	SCCL-7	164.640	0.346	0.676	0.460
8	SCCL-8	123.400	0.501	0.318	0.395
9	NCL-1	178.950	0.388	0.787	0.506
10	MCL-1	180.720	0.299	0.998	0.461
11	MCL-2	178.310	0.385	0.951	0.518
12	MCL-3	166.550	0.361	0.519	0.414
13	MCL-4	163.900	0.451	0.768	0.538
14	MCL-5	162.560	0.438	0.452	0.442

15	MCL-6	184.950	0.557	1.582	0.823
16	MCL-7	161.370	0.649	1.165	0.819
17	SECL-1	123.620	0.907	0.531	0.721
18	SECL-2	113.610	1.366	0.541	0.857
19	SECL-3	127.050	0.520	0.252	0.385
20	SECL-4	342.310	0.007	4.563	0.472
21	SECL-5	167.250	0.090	0.241	0.135
22	SECL-6	133.750	0.504	0.328	0.420
23	ECL-1	157.510	0.498	1.023	0.685

4.3.3 Wet oxidation potential analysis

The coal molecule may consist of two parts: the aliphatic or hydro-aromatic structure that are more prone to oxidation and condensed aromatic structure, which are resistant to oxidation. Hydroxyl group in the aromatic structure gets oxidized faster and gives a very high degree of reactivity to coal. Thus, low rank coals are rather easily oxidized due to the above and also due to smaller degree of condensation of aromatic structures in them. Lower rank coals on oxidation produce large amounts of aliphatic acids compared to higher rank coals, because low rank coals contain more branched aliphatic hydro-carbons. Since the high rank coals have structure close to that of graphite, it is less liable to oxidation and the products contain more aromatics than aliphatics. It is generally seen that lower the potential difference, lower is the susceptibility of coal to spontaneous heating. Figure 4.8 shows the photographic view of the wet oxidation apparatus.

It is based on the chemical reaction of coal sample with solution of $KMnO_4$ and KOH. The solution of alkaline potassium permanganate and potassium hydroxide with coal sample forms an electrochemical cell which on stirring produces EMF against a standard potential of 0.56V. The plot of the EMF‘s versus time gives an idea of the susceptibility of the coal sample towards spontaneous heating (Tarafdar and Guha, 1989).

Figure 4.8: Photographic view of the Experimental Setup of Wet Oxidation Potential Apparatus

4.3.3.1 Experimental Procedure

The beaker along with the electrodes was placed over a magnetic stirrer such that a homogeneous mixture of coal and alkali solution is maintained. The teflon coated fish of the magnetic stirrer was placed inside the beaker. 0.5 g of coal sample of –212μ (micron)size was mixed with 100 ml of deci-normal solution of potassium permanganate ($KMnO_4$) in 1N potassium hydroxide (KOH) solution in a beaker and the coal sample was subjected to wet oxidation process. The coal-oxidant suspension was continuously stirred using the magnetic stirrer. The potential difference (EMF) was recorded between the calomel and carbon electrodes over a period of time by using a millivolt meter till the potential difference attained a nearly constant value. Different samples require different time duration for attaining a nearly constant potential difference (ΔE). It was observed that almost all the coal samples attained a constant value after 30 minutes. Therefore this time period was chosen to find out the wet oxidation potential difference for a comparative study. The results are presented in Table 4.7.

Table4.7: Wet oxidation potential difference of coal samples

Sl. No.	Sample Name	WET OXIDATION POTENTIAL (mV)
1	SCCL-1	94.7
2	SCCL-2	127.6
3	SCCL-3	120.5
4	SCCL-4	124.2
5	SCCL-5	85.8
6	SCCL-6	88.3
7	SCCL-7	101.7
8	SCCL-8	107
9	NCL-1	92.4
10	MCL-1	114.7
11	MCL-2	115
12	MCL-3	121
13	MCL-4	110.9
14	MCL-5	102.3
15	MCL-6	93
16	MCL-7	126
17	SECL-1	98.3
18	SECL-2	104
19	SECL-3	116.8
20	SECL-4	110.3
21	SECL-5	122.4
22	SECL-6	115.3
23	ECL-1	93

CHAPTER 6

CORRELATION STUDY

CHAPTER 5

5. CORRELATION STUDY

In order to establish the effects of intrinsic properties on the spontaneous combustion susceptibility of coal samples, correlation studies were carried out. The correlation coefficients between the intrinsic properties and susceptibility indices were found out taking intrinsic properties, viz. moisture, volatile matter, ash and gross calorific value as independent variable and the susceptibility indices determined by CPT, DTA and wet oxidation potential analyses as dependent variable. Empirical relations were also established between them and curves were plotted taking independent variables in horizontal axis and dependent variables on vertical axis. The correlation coefficients between CPT and intrinsic properties are presented in table 5.1 and the correlation plots have been presented in figures 5.1 to 5.4. The correlation coefficients between T_c, IIA, IIB and overall slope of stage II and the intrinsic properties are presented in tables 5.2 to 5.5, and the correlation plots have been presented in figures 5.5 to 5.20. Similarly, the correlation coefficients between wet oxidation potential difference (mV) and the intrinsic properties are presented in table 5.6, and the correlation plots have been presented in figures 5.21 to 5.24.

Table5.1: Correlation between CPT and intrinsic properties

Sl. No.	Independent variable	Empirical relation	r
1	M	$\mathrm{CPT} = 227.69 - 48.03\mathrm{M} + 12.398\mathrm{M}^2 - 1.24\mathrm{M}^3$	0.717
2	VM	$\mathrm{CPT} = 161.155\mathrm{VM}^{0.286/\mathrm{VM}}$	0.127
3	A	$\mathrm{CPT} = 178.35 - 1.5\mathrm{A} + 0.05\mathrm{A}^2 - 0.0005\mathrm{A}^3$	0.16
4	CV	$\mathrm{CPT} = 158.89\ \mathrm{CV}^{1.14\mathrm{CV}}$	0.264

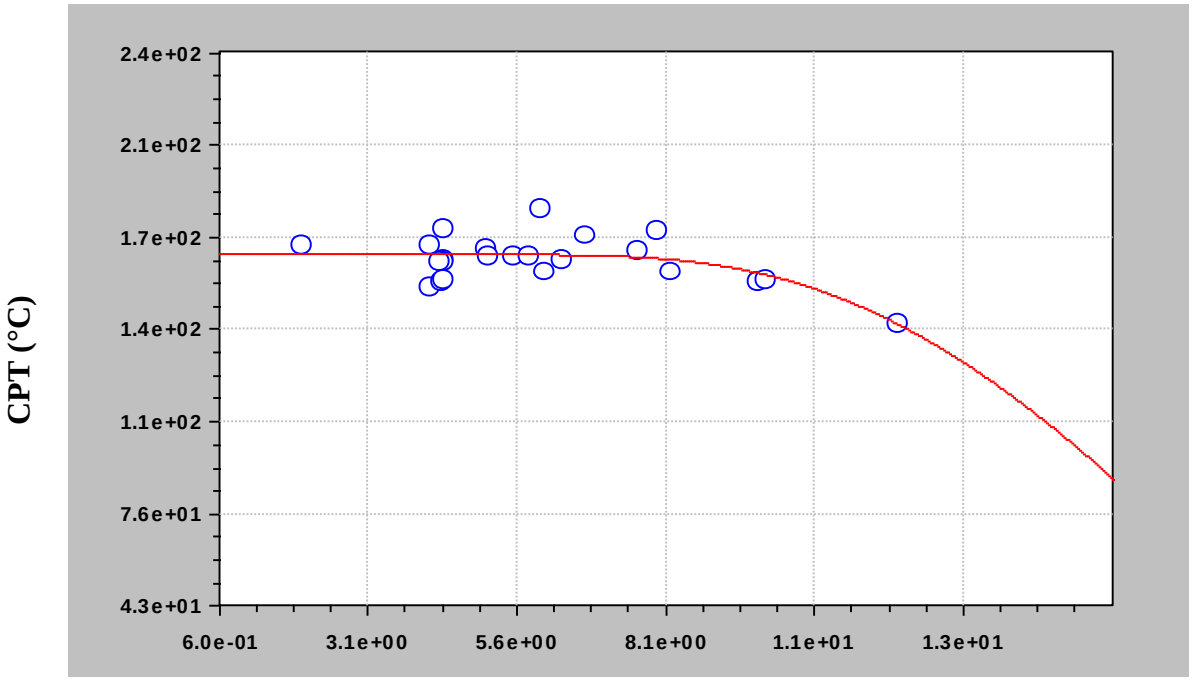


Figure 5.1: Correlation plot between CPT and moisture content

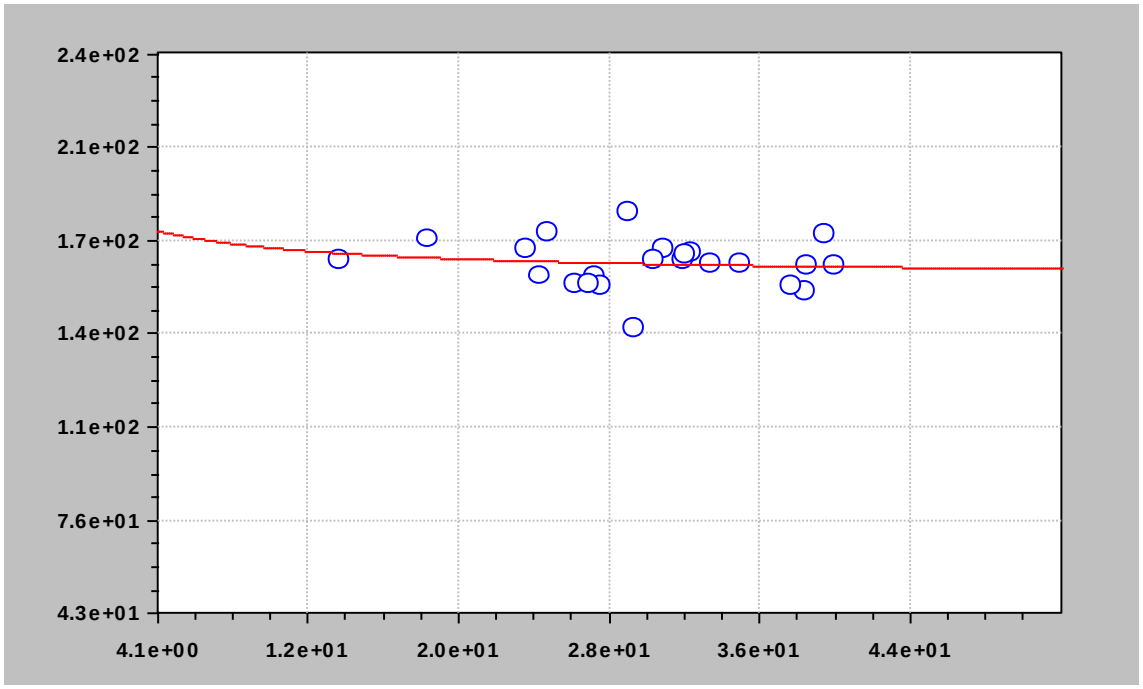


Figure 5.2: Correlation plot between CPT and volatile matter

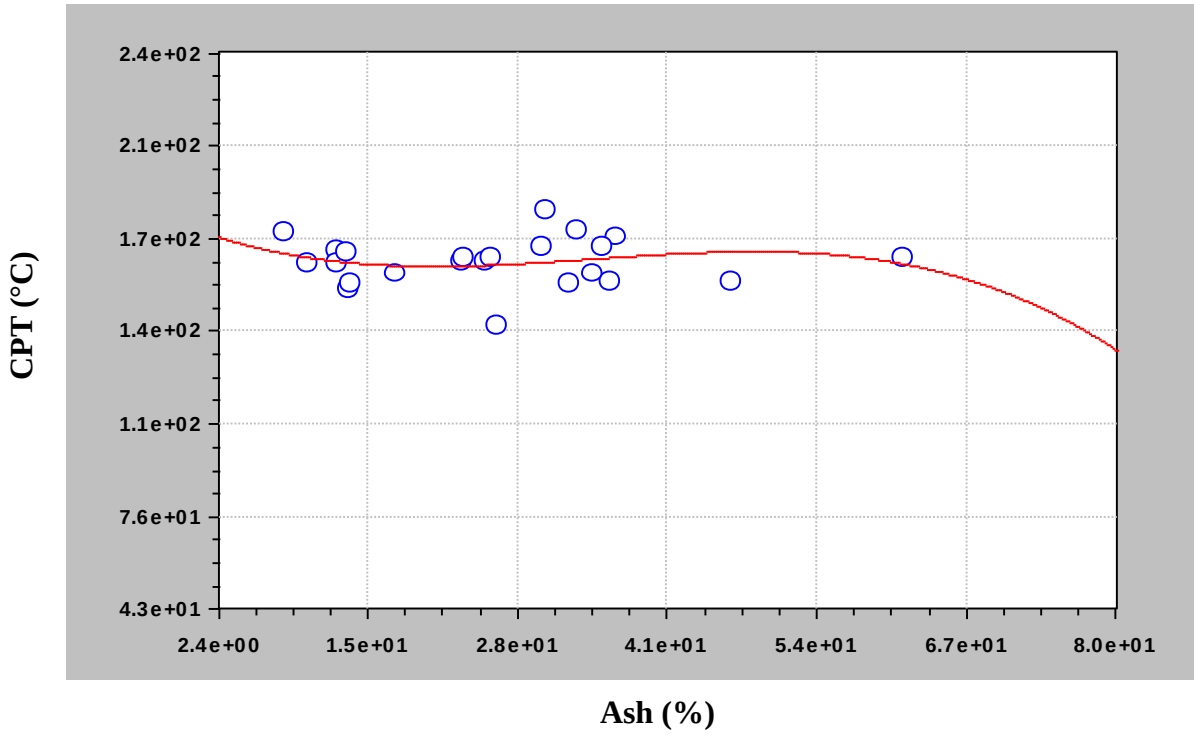


Figure5.3: Correlation plot between CPT and ash content

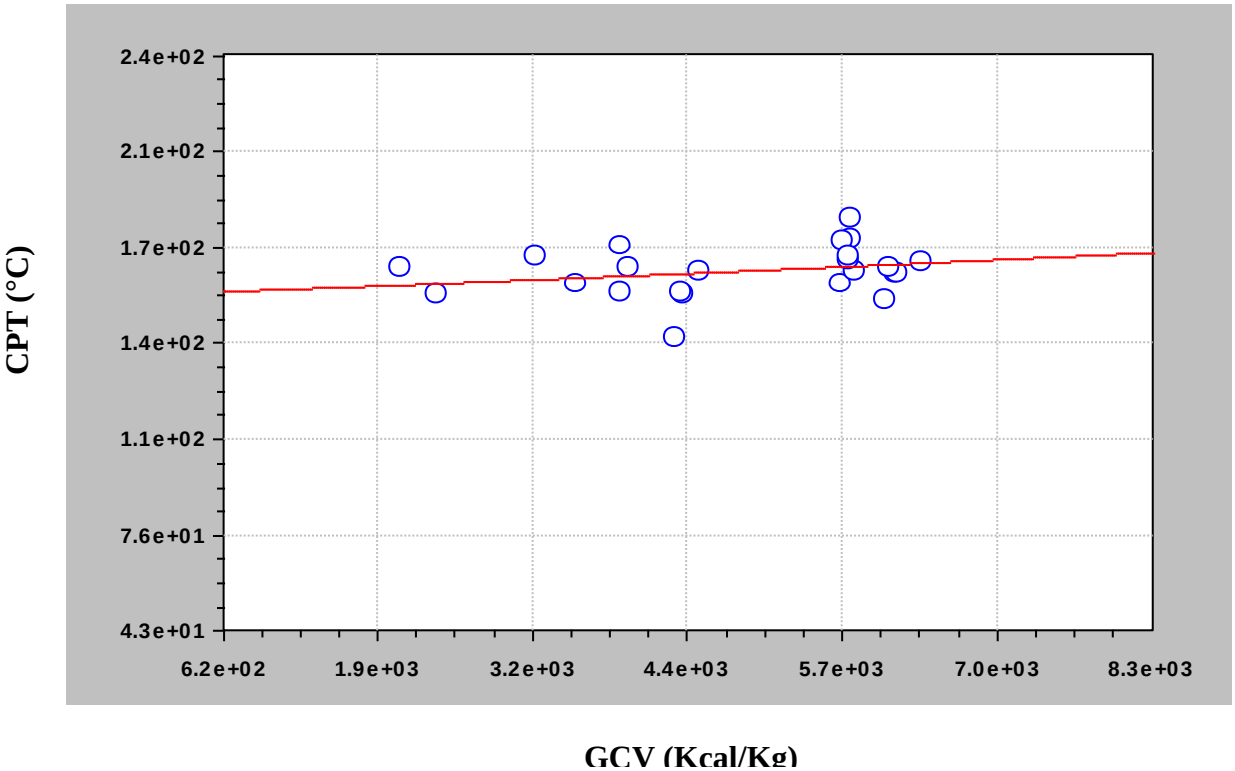


Figure 5.4: Correlation plot between CPT and gross calorific value

Table 5.2: Correlation between T_c of DTA thermogram and intrinsic properties

Sl. No.	Independent variable	Empirical relation	r
1	M	$T_c = 184.33 - 1766.15e^{-10.48M^{-0.47}}$	0.380
2	VM	$T_c = e^{-9.404+\frac{87.32}{VM}+3.375\ln VM}$	0.880
3	A	$T_c = 416.72 - 278.61e^{-0.00014A^{10.94}}$	0.533
4	CV	$T_c = 488.166 - 0.1498CV + 1.60CV^2$	0.586

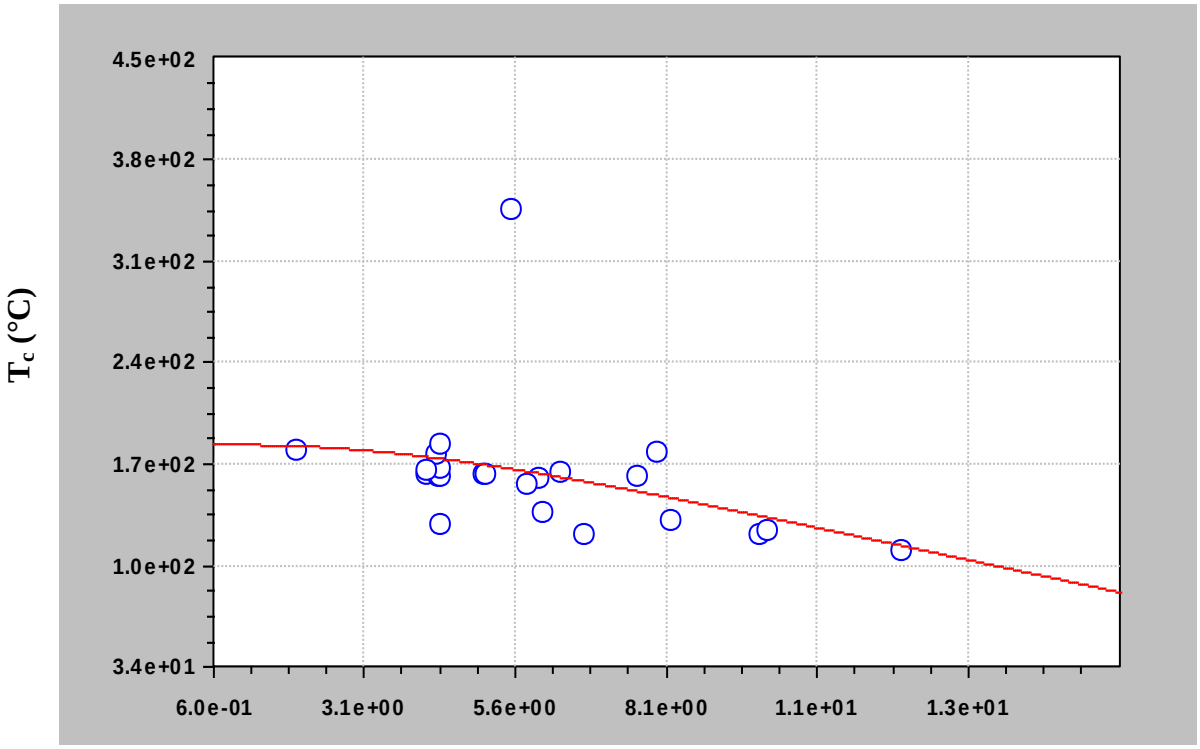


Figure5.5: Correlation plot between T_c of DTA thermogram and moisture

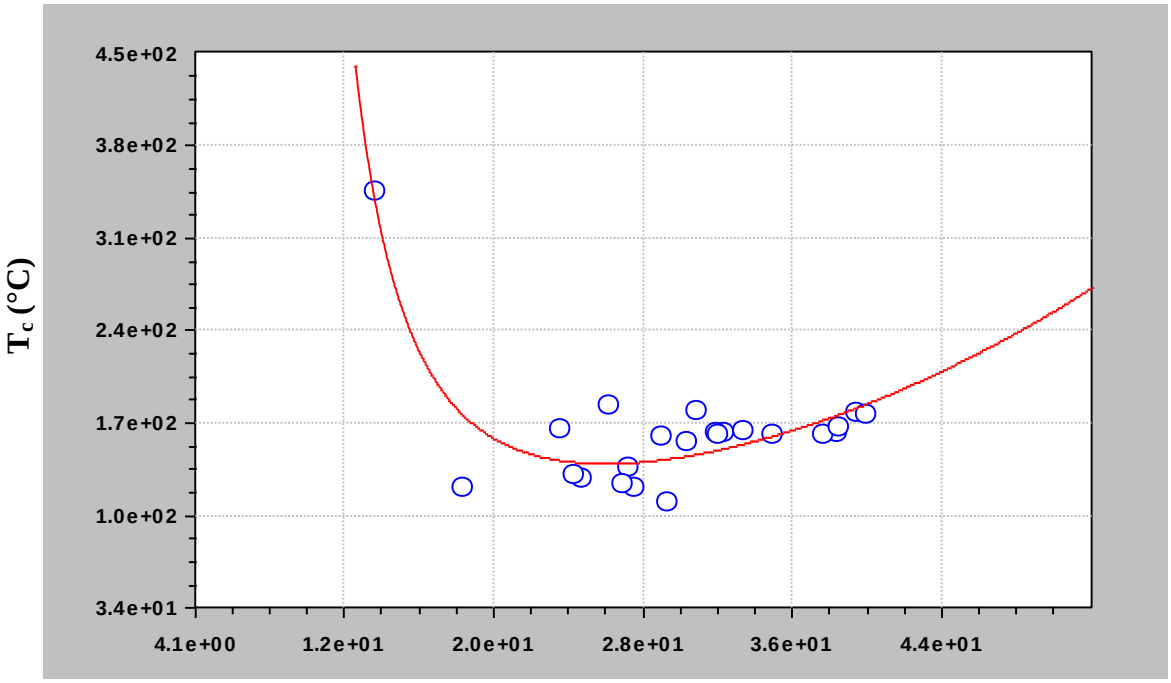


Figure5.6: Correlation plot between T_c of DTA thermogram and volatile matter

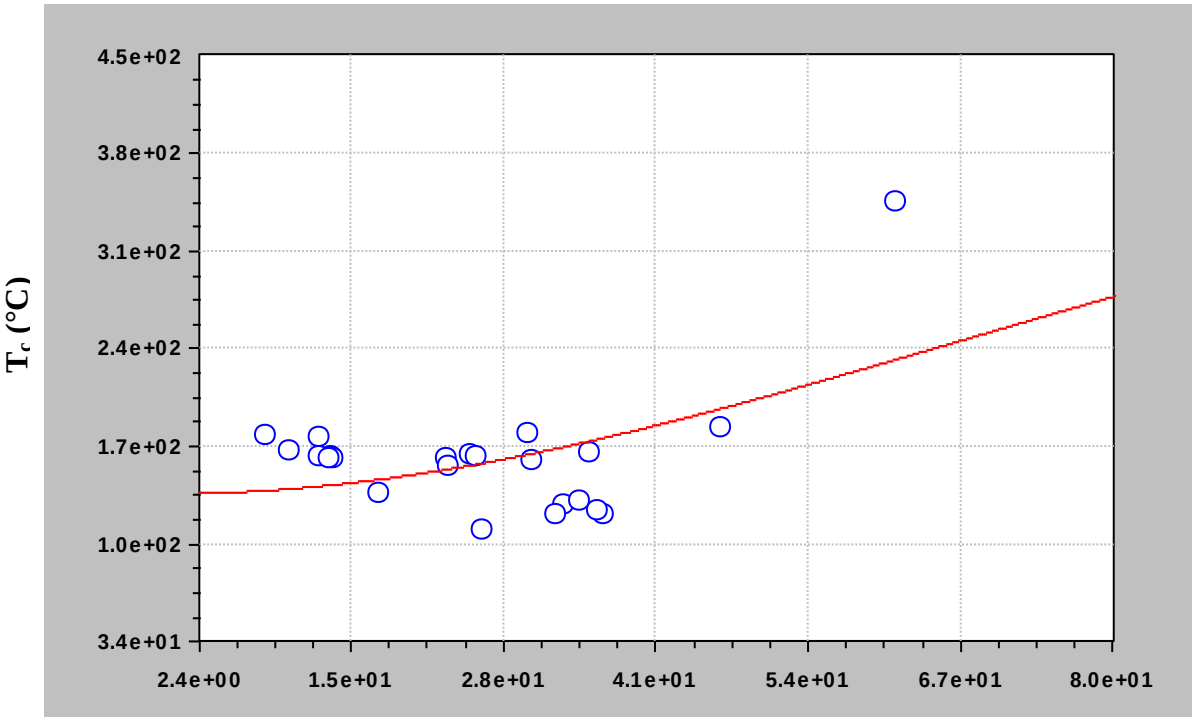


Figure5.7: Correlation plot between onset temperature (T_c) of DTA thermogram and ash content

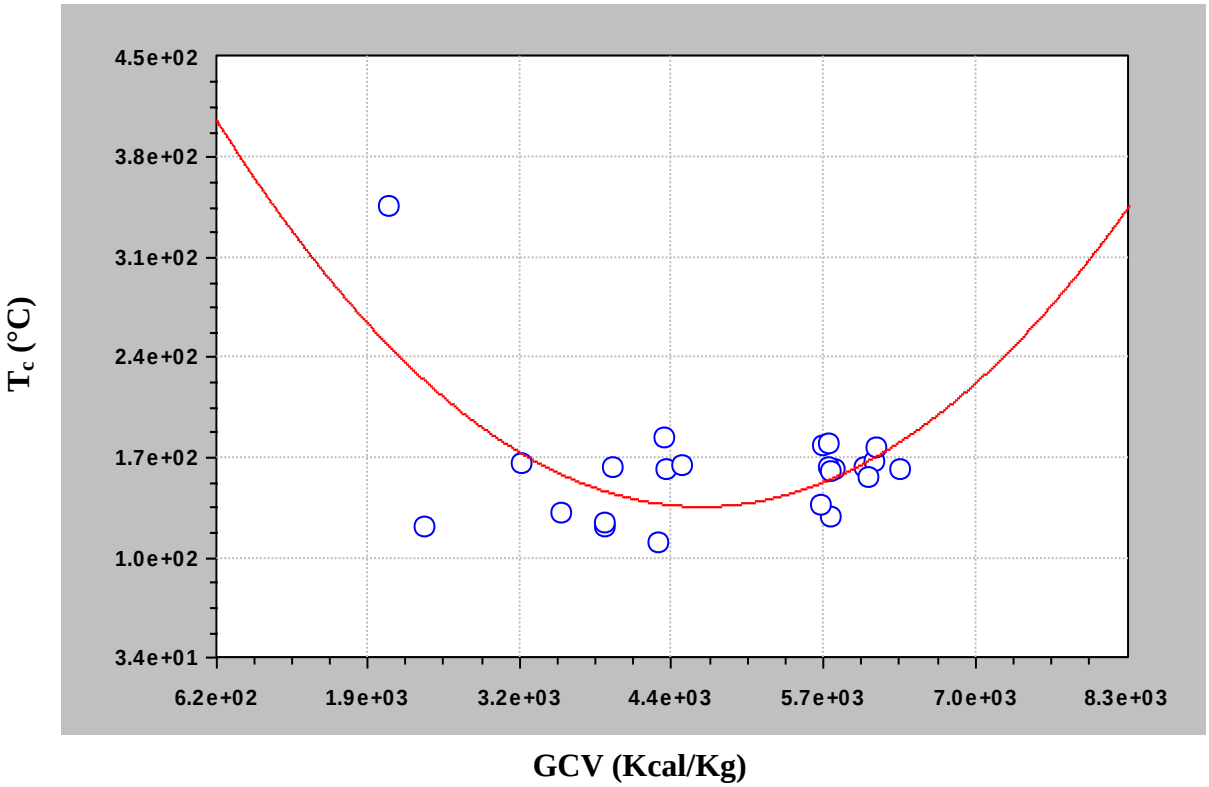


Figure5.8: Correlation plot between T_c of DTA thermogram and gross calorific value

Table5.3: Correlation between average slope of stage II A of DTA thermogram and intrinsic properties

Sl. No.	Independent variable	Empirical relation	r
1	M	$\mathrm{IIA} = \frac{1}{2.87 + 8.43e^{-5}\mathrm{M} - 0.015\mathrm{M}^2}$	0.804
2	VM	$\mathrm{IIA} = \frac{-0.0033 + 0.0023\mathrm{VM}}{1 - 0.042\mathrm{VM} + 0.00055\mathrm{VM}^2}$	0.503
3	A	$\mathrm{IIA} = 0.16 + 0.029\mathrm{A} - 0.0005\mathrm{A}^2$	0.462
4	CV	$\mathrm{IIA} = 0.1184\mathrm{CV}^{3.0089\mathrm{CV}}$	0.628

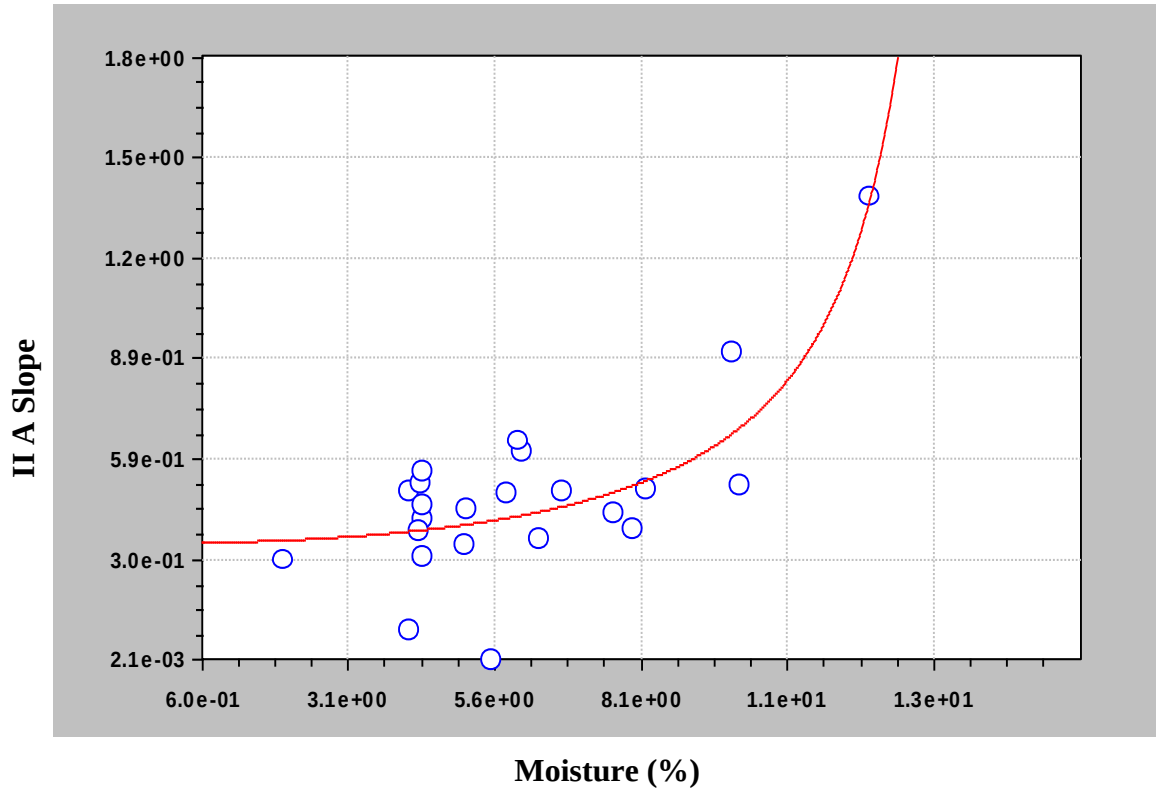


Figure5.9: Correlation plot between average slope of stage II A of DTA thermogram and Moisture content

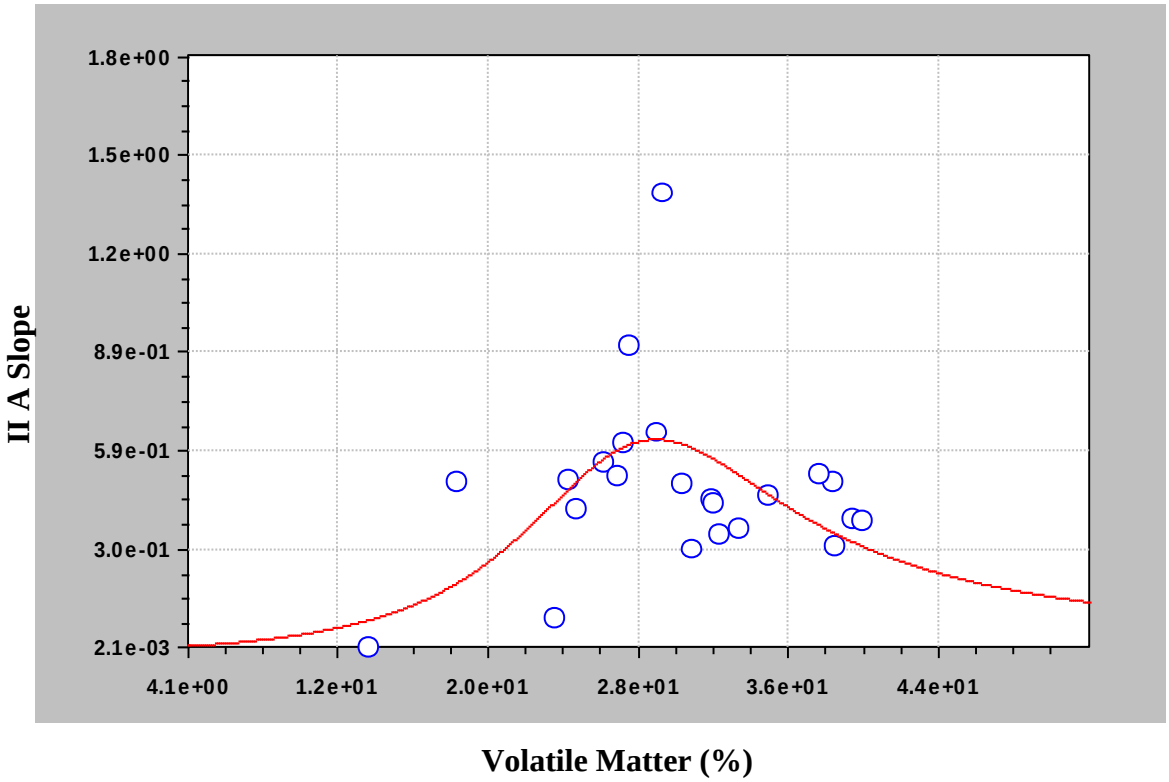


Figure5.10: Correlation plot between average slope of stage II A of DTA thermogram and volatile matter

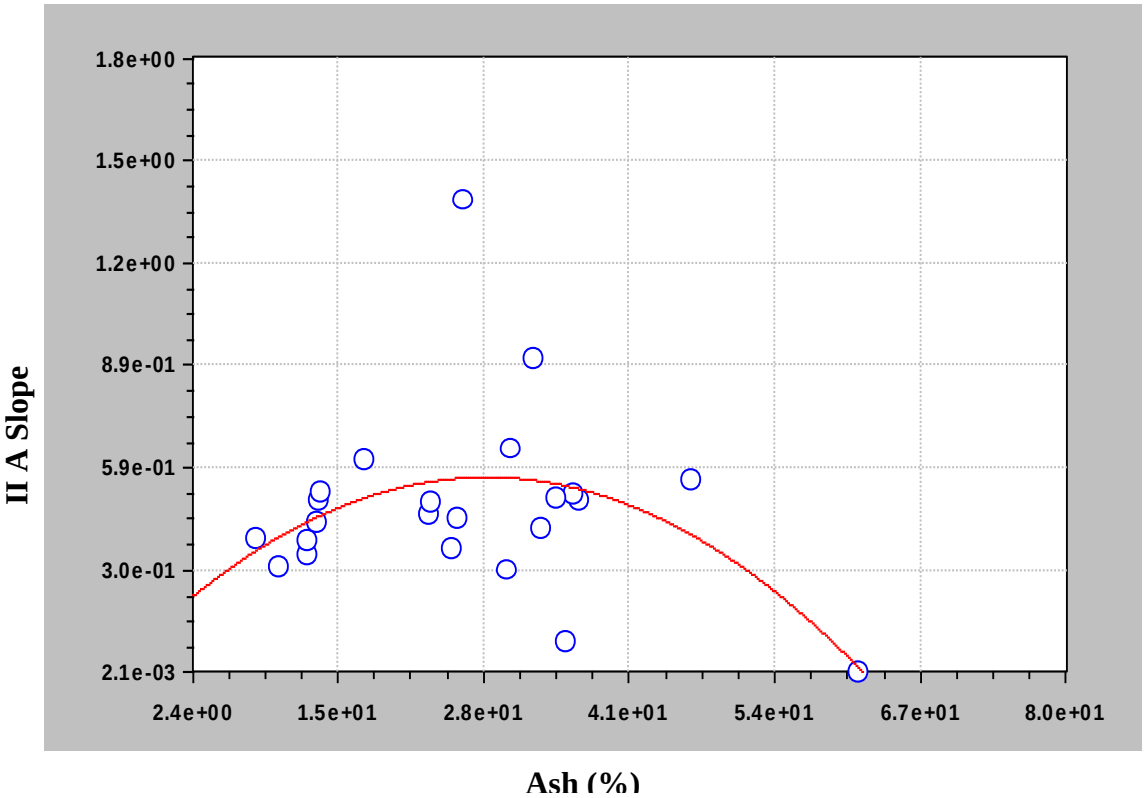


Figure5.11: Correlation plot between average slope of stage II A of DTA thermogram and ash content

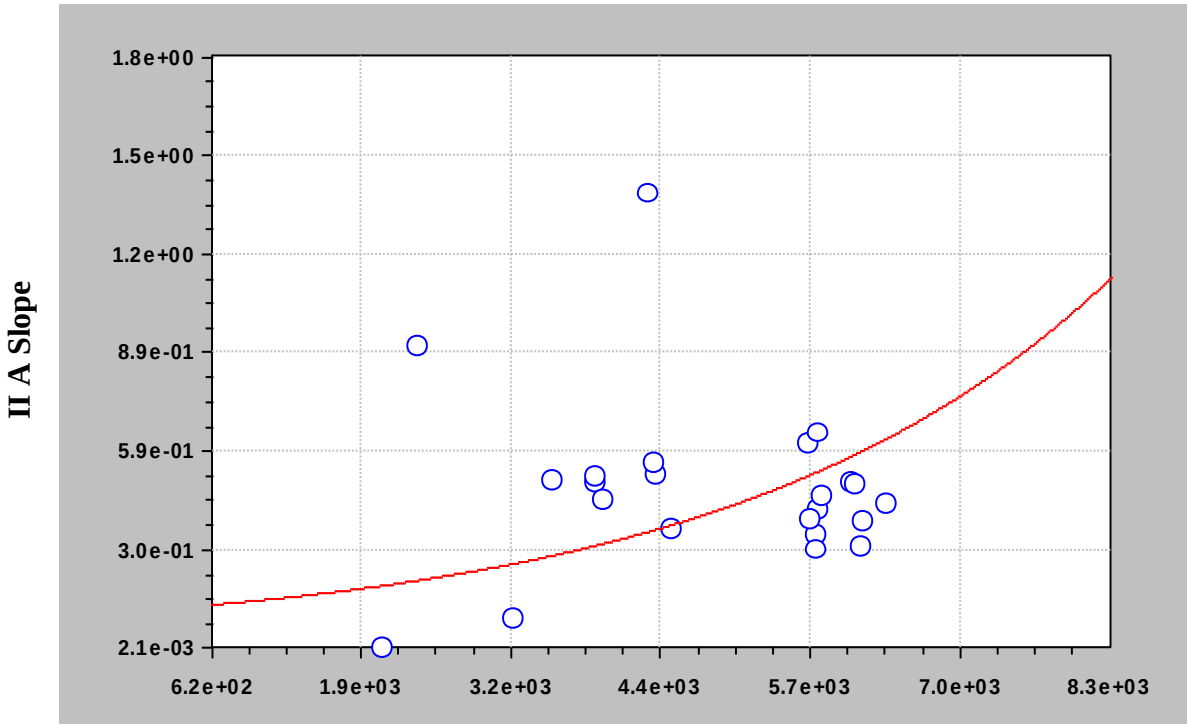


Figure5.12: Correlation plot between average slope of stage II A of DTA thermogram and gross calorific value

Table 5.4: Correlation between average slope of stage II B of DTA thermogram and intrinsic properties

Sl. No.	Independent variable	Empirical relation	R
1	M	$IIB = \frac{0.059 - 0.008M}{1 - 0.357M + 0.032M^2}$	0.701
2	VM	$IIB = \frac{0.376VM}{-12.56 + VM}$	0.87
3	A	$IIB = 5.62 - 5.32e^{-0.0002A^{1.95}}$	0.624
4	CV	$IIB = \frac{0.336 * CV}{-1915.17 + CV}$	0.822

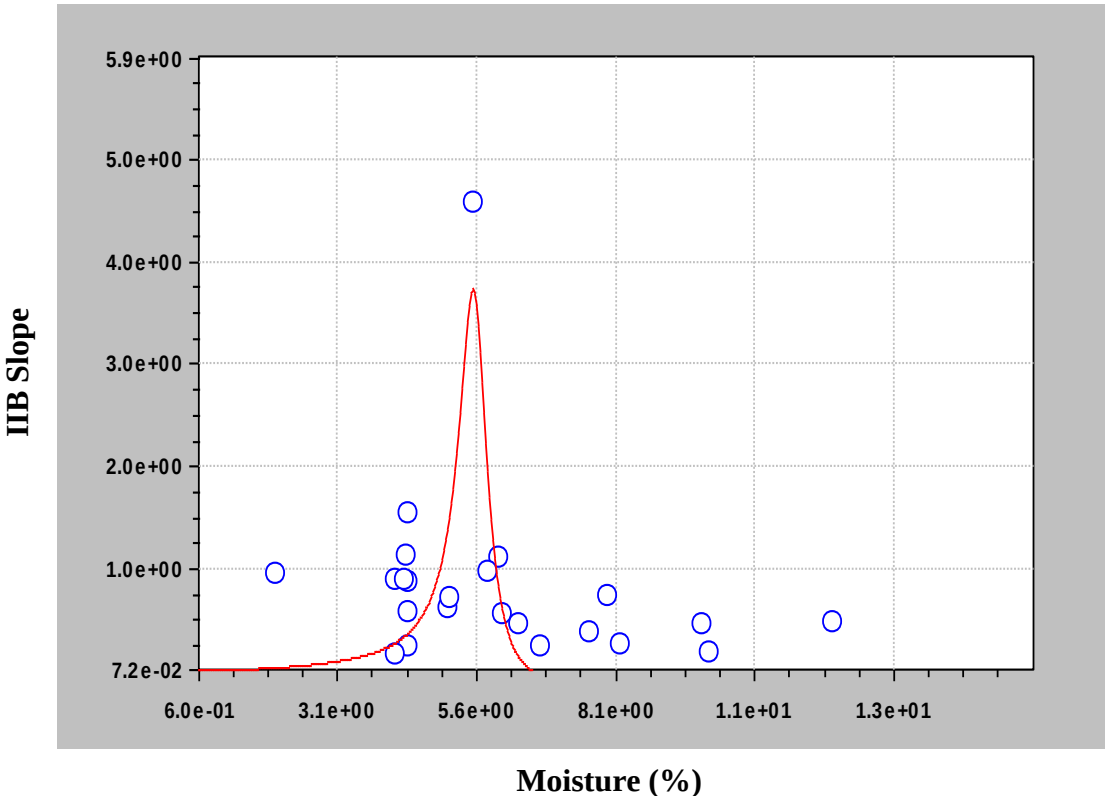


Figure5.13: Correlation plot between average slope of stage IIB of DTA thermogram and moisture content

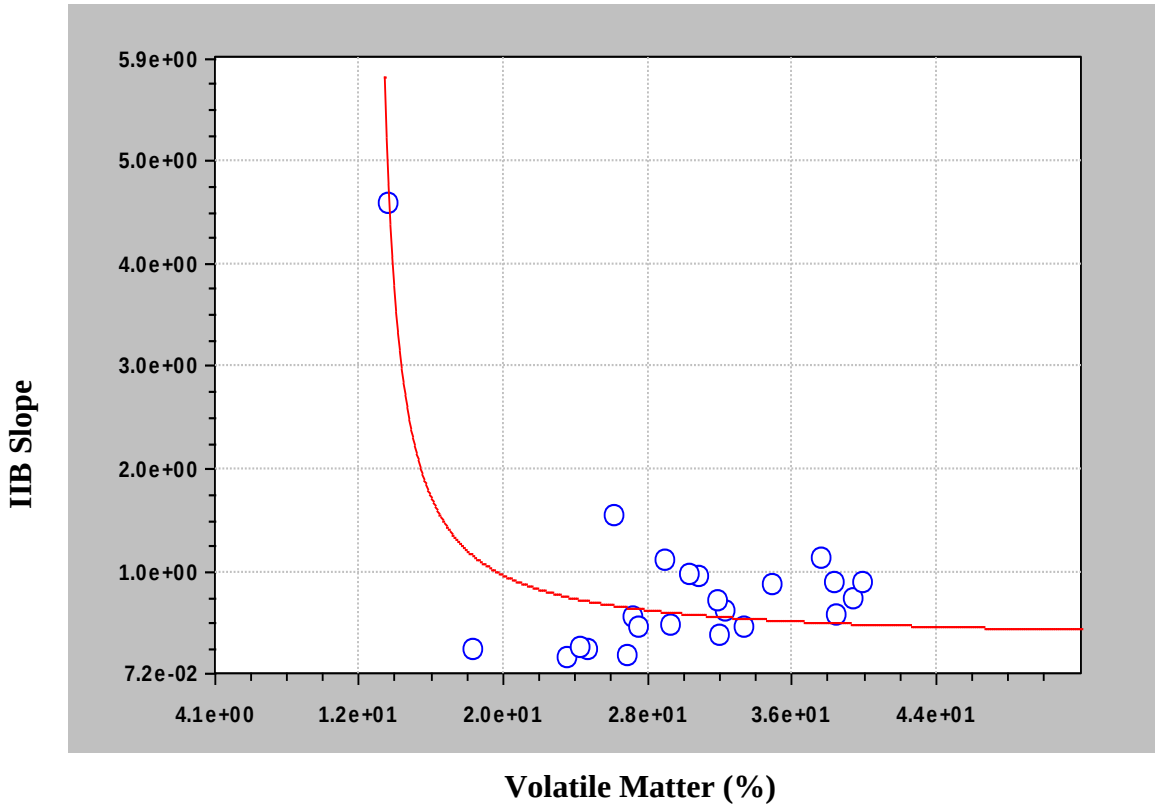


Figure5.14: Correlation plot between average slope of stage IIB of DTA thermogram and volatile matter

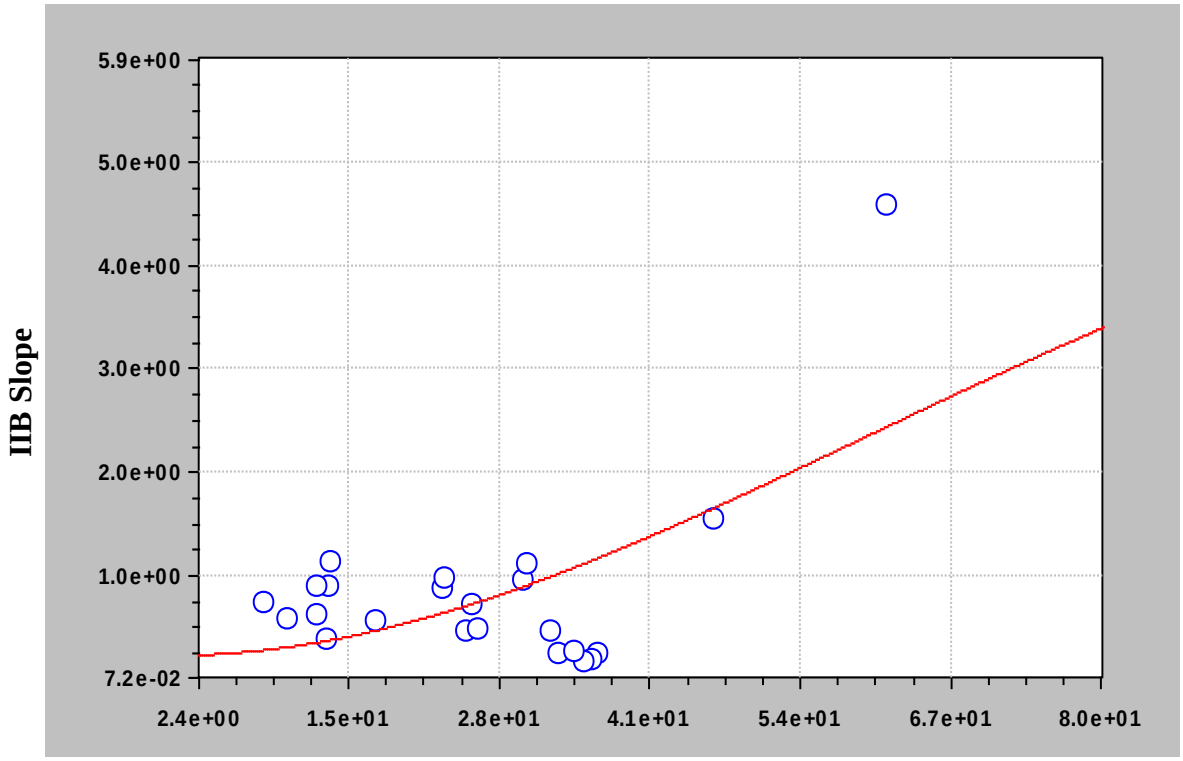


Figure 5.15: Correlation plot between average slope of stage IIB of DTA thermogram and ash content

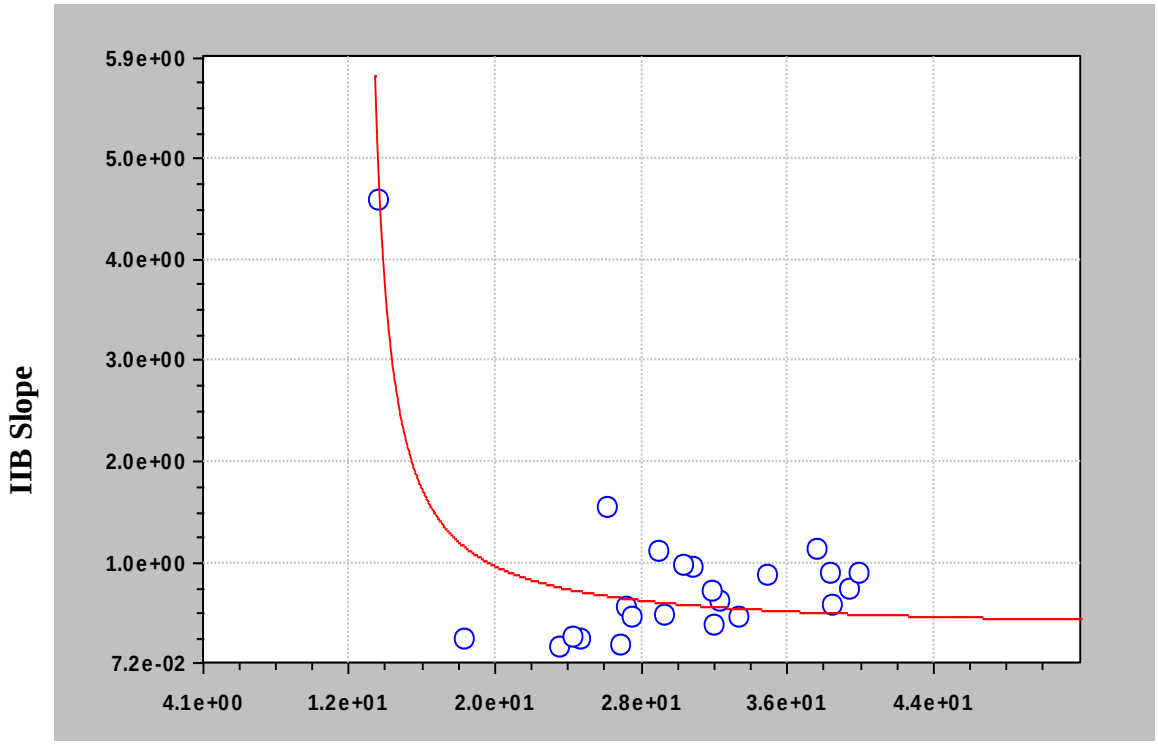


Fig. 6.16: Correlation plot between average slope of stage IIB of DTA thermogram and gross calorific value

Table5.5: Correlation between average slope of stage II of DTA thermogram and intrinsic properties

Sl. No.	Independent variable	Empirical relation	R
1	M	$II = 0.321 - 0.048M + 0.014M^2 - 0.004M^3$	0.416
2	VM	$II = \frac{0.23 - 0.003VM}{1 - 0.042VM + 0.00055VM^2}$	0.320
3	A	$II = e^{1.77 - \frac{10.73}{A} - 0.59\ln A}$	0.210
4	CV	$II = 0.275 + 0.00011CV - 1.079CV^2$	0.143

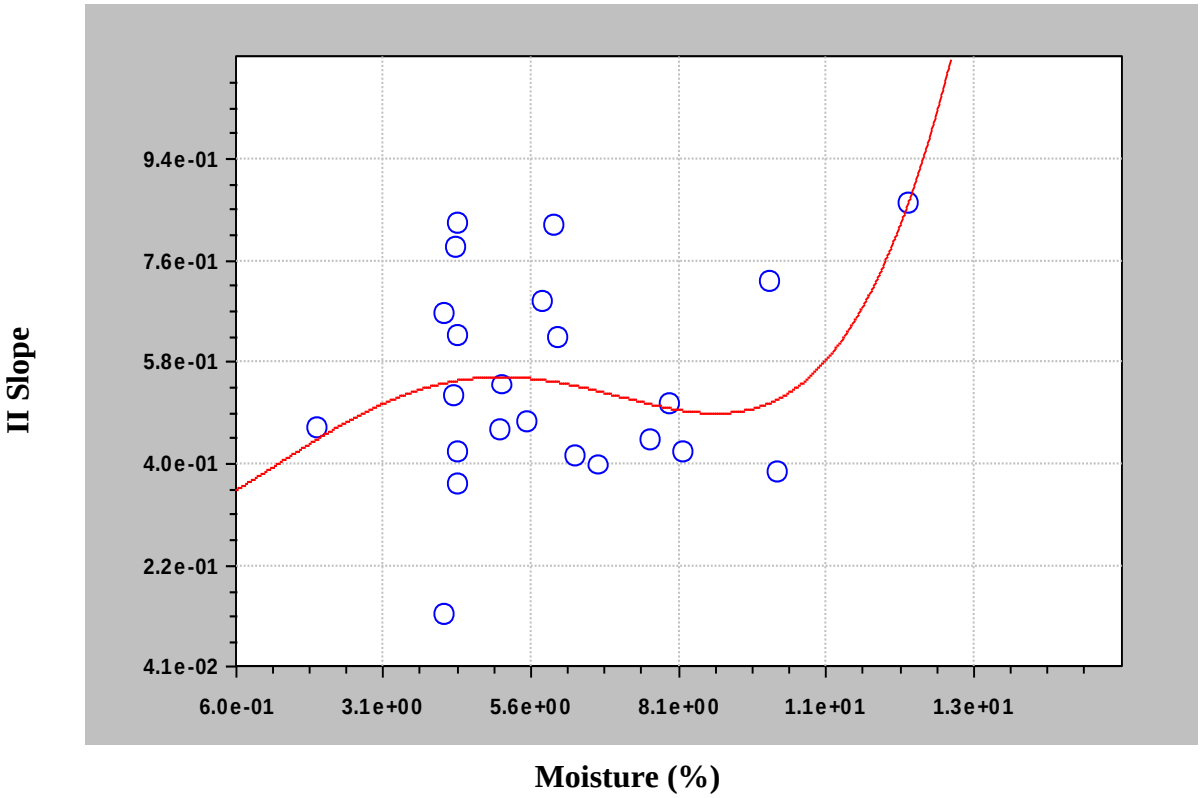


Figure5.17: Correlation plot between average slope of stage II of DTA thermogram and Moisture content

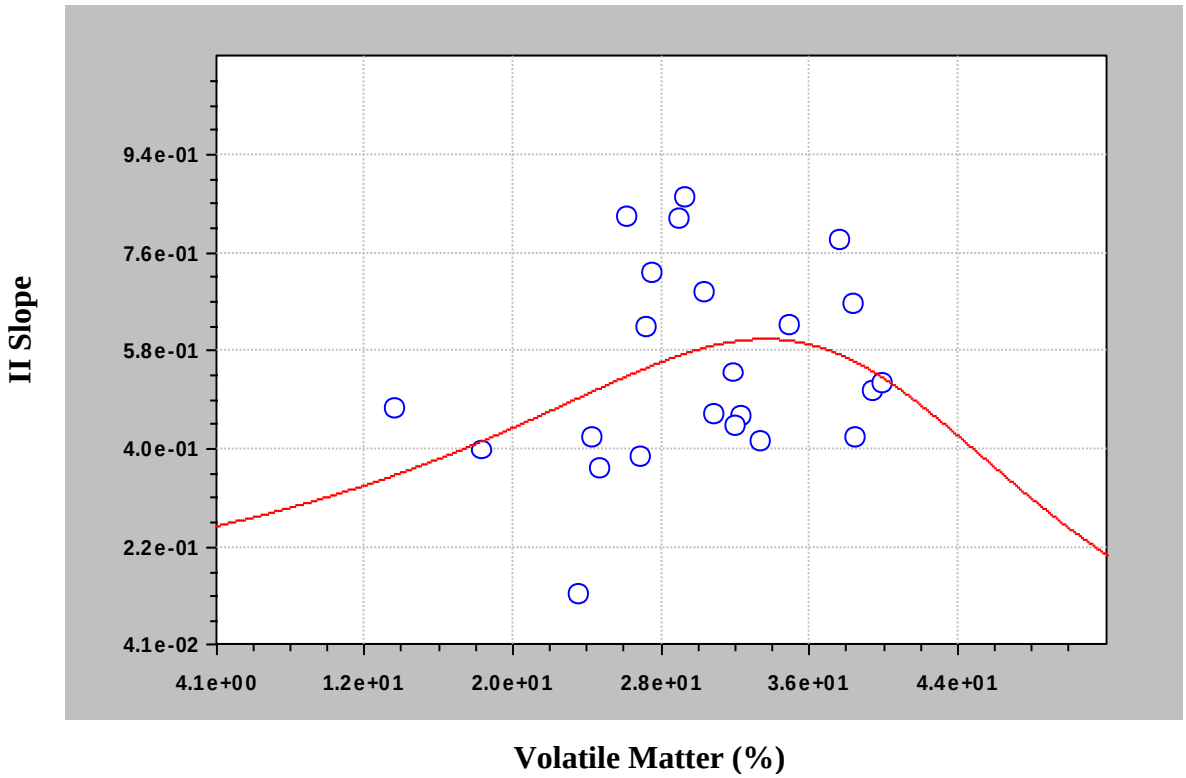


Figure5.18: Correlation plot between average slope of stage II of DTA thermogram and volatile matter

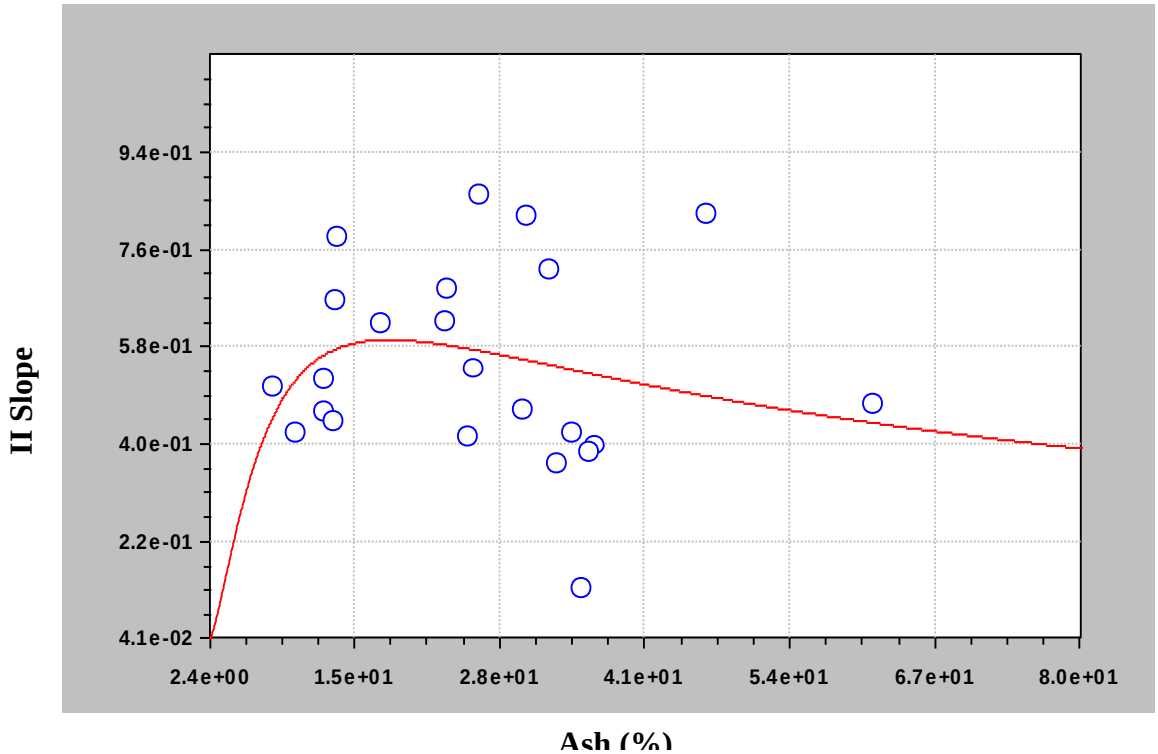


Figure5.19: Correlation plot between average slope of stage II of DTA thermogram and ash content

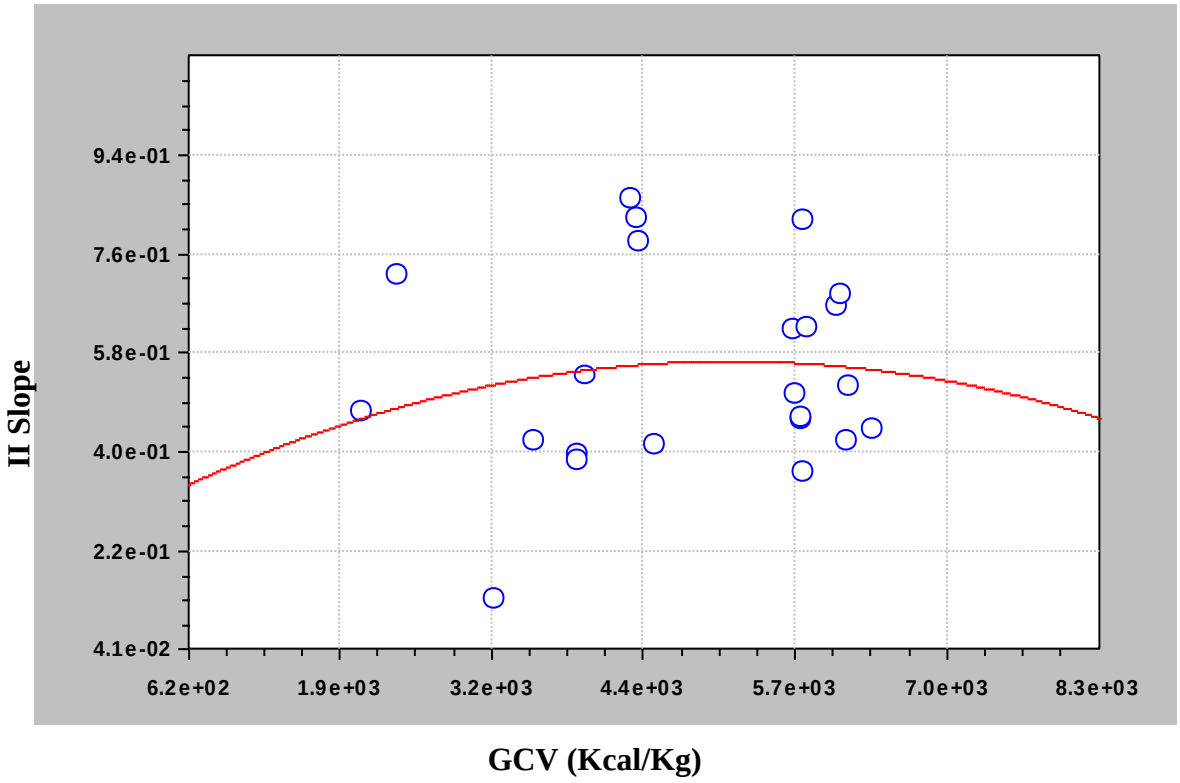


Fig. 6.20: Correlation plot between average slope of stage II of DTA thermogram and gross calorific value

Table5.6: Correlation between wet oxidation potential difference (WOPD) and intrinsic properties

Sl. No.	Independent variable	Empirical relation	R
1	M	$\text{WOPD} = \frac{3.789 + 2.95M}{1 + 2.79M - 155M^2}$	0.087
2	VM	$\text{WOPD} = 122.88\text{VM}^{-0.0013\text{VM}}$	0.308
3	A	$\text{WOPD} = 111.05(1 - \text{e}^{-0.219\text{A}})$	0.394
4	CV	$\text{WOPD} = 77.34 + 0.018\text{CV} - 2.285\text{CV}^2$	0.337

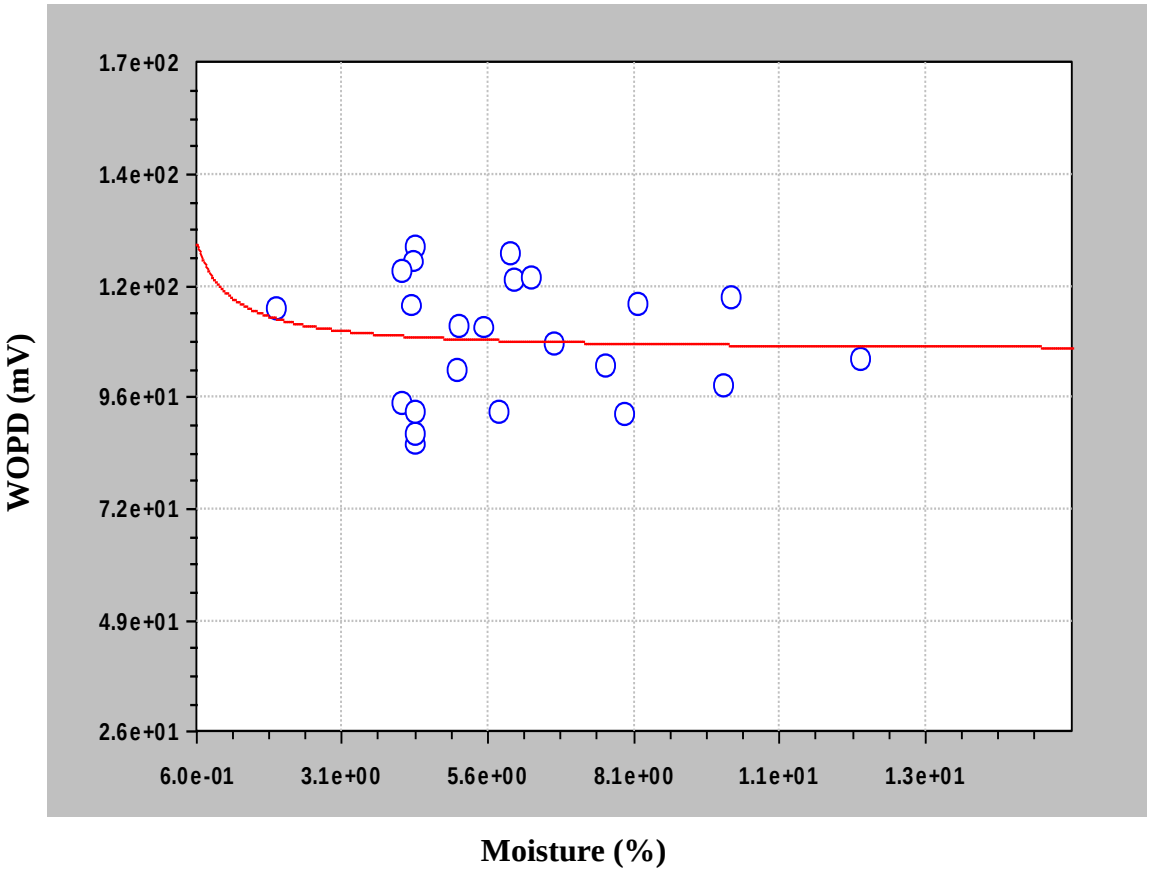


Figure 5.21: Correlation plot between WOPD and Moisture content

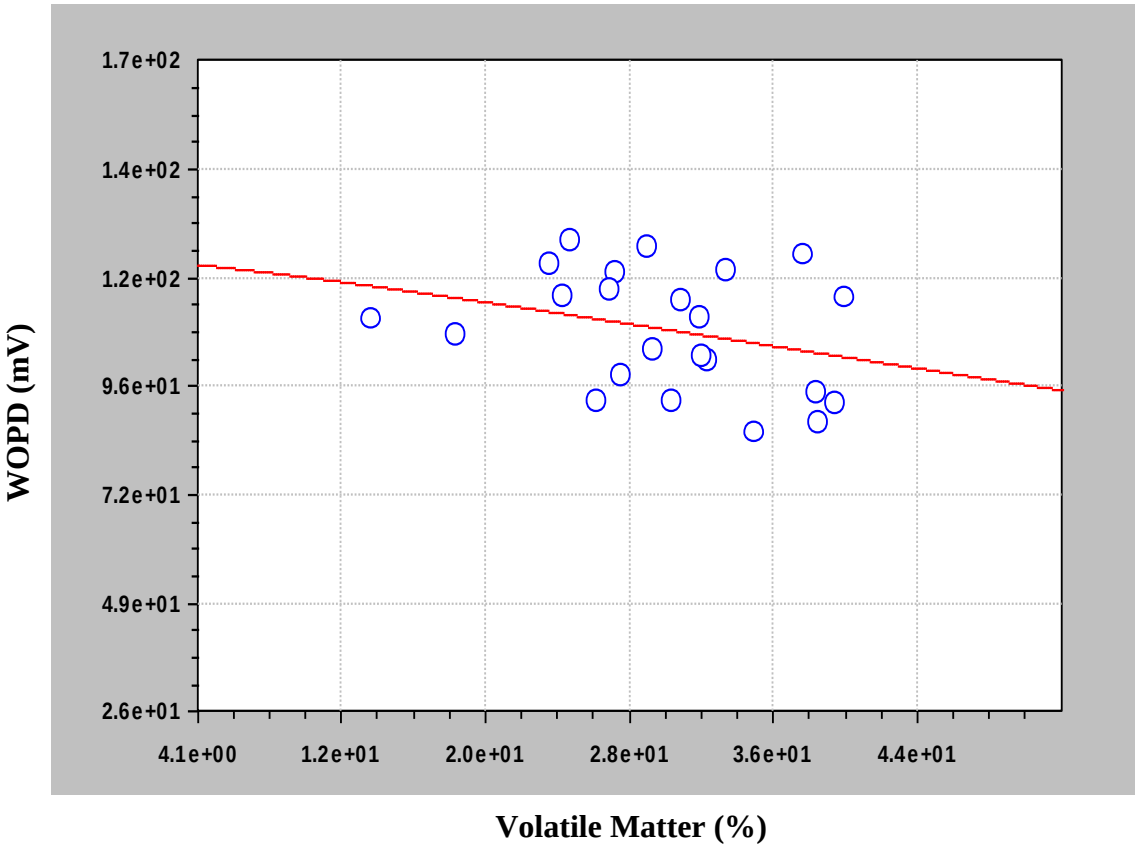


Figure5.22: Correlation plot between WOPD and volatile matter

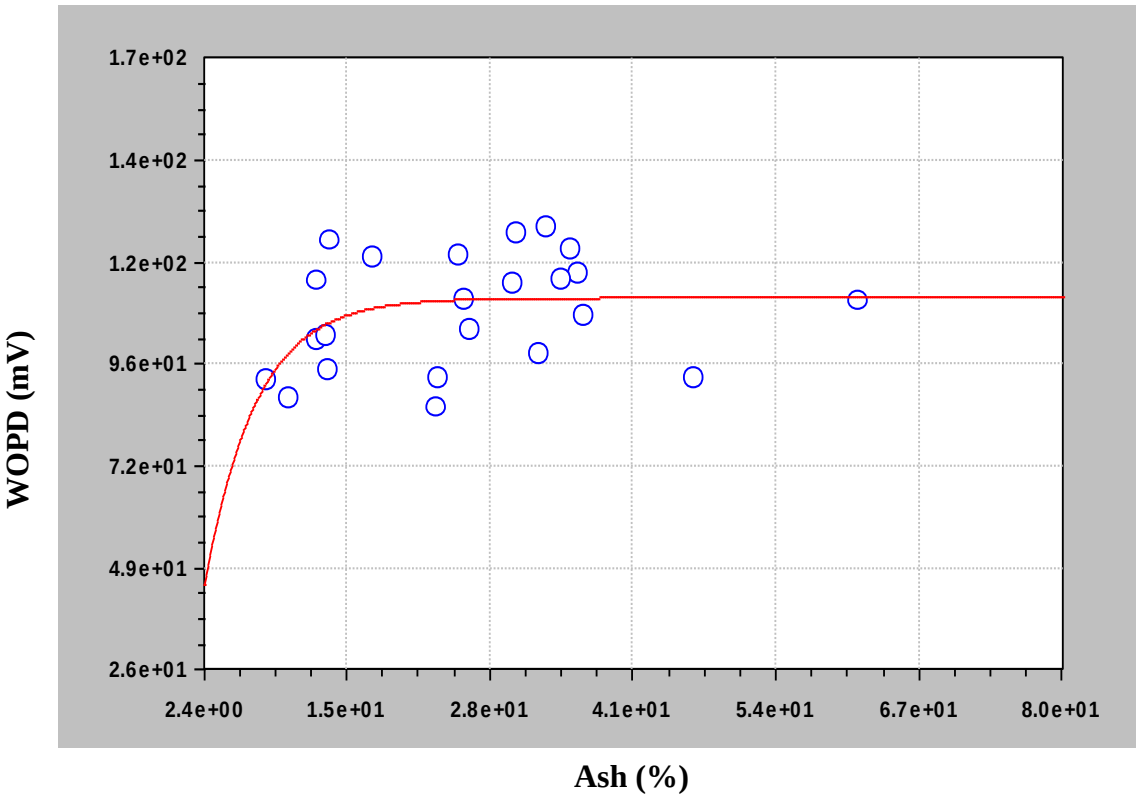


Figure5.23: Correlation plot between WOPD and Ash content

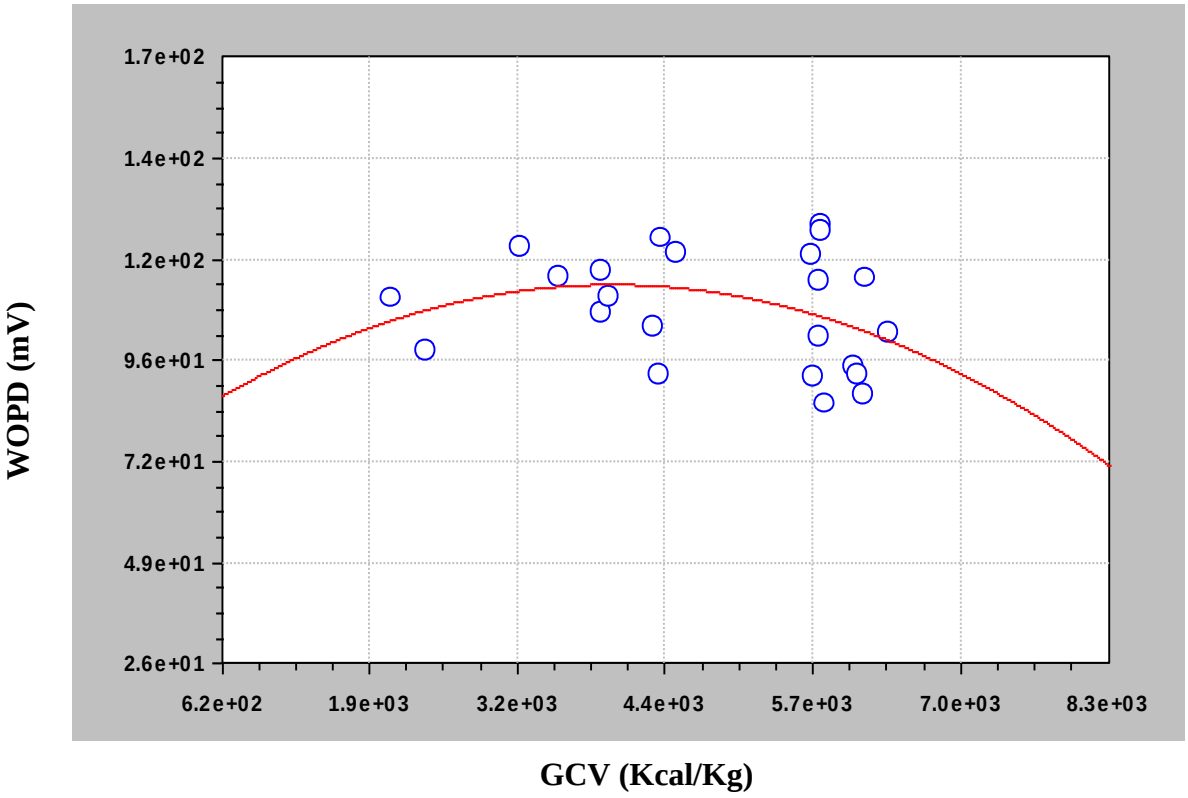


Figure5.24: Correlation plot between WOPD and Gross Calorific Value

CHAPTER 6

DISCUSSION AND CONCLUSION

CHAPTER 6

6. DISCUSSION AND CONCLUSION

6.1 DISCUSSION

A total of 23 coal samples from different coal fields of India were collected and analyzed for their spontaneous heating susceptibility. Of these, 8 were from SCCL, 1 from NCL, 7 from MCL, 6 from SECL and 1 from ECL. The experimental investigations of their intrinsic properties and susceptibility indices, and correlation analysis between them are discussed in the following sections.

The moisture content of the coal samples varied from 2 %(MCL-1) to 12.05 % (SECL-2). It was observed that the coal samples from SECL had higher moisture content than others followed by those from NCL and MCL. It was observed that samples of higher moisture content were more susceptible to spontaneous heating.

The volatile matter of the coal samples varied from 13.7 % (SECL-4) to 39.84 (MCL-2). It was found that the SECL coal samples had lower volatile matter than others. The MCL coal samples had notably high volatile matter content than SCCL and SECL coal samples. The ash content of the coal samples varied between 8 % (NCL-1) to 61.34 % (SECL-4). The SECL coal samples showed comparatively higher ash content than others.

The gross calorific values varied between 2073.66 Kcal/kg (SECL-4) and 6351.89 Kcal/kg (MCL-5). The SECL coal samples had comparatively lower values than others.

The CPT value was the least in the coal sample SECL-2 (144°C) indicating that it was more susceptible than others. A total of 5 samples (SECL-1, SECL-2, SECL-3, MCL-6 and SCCL-1) were found to have CPT values ranging from 140-160°C indicating that they were moderately susceptible. The rest of the samples had values greater than 160°Cindicating that they were poorly susceptible.

Transition temperature (T_c) was lowest in the case of the coal sample SECL-2 indicating that it was more susceptible to spontaneous heating than others. In earlier work (Panigrahi and Sahu, 2004), it was found that the coal samples having T_c values between 122-140 °C were highly susceptible to spontaneous heating. In this work, it may be considered that the

coal samples having T_c values below than 140 °C (SECL-1, SECL-2, SECL-3, SECL-6 and SCCL-8) are highly susceptible to spontaneous heating.

The average slopes of stage II A varied between 0.007 (SECL-4) to 1.366 (SECL-2). IIB slope values varied between 0.241 (SECL-5) to 1.582 (MCL-6). The overall average values of stage II varied between 0.135 (SECL-5) to 0.857 (SECL-2).

The wet oxidation potential values ranged between 85.8 mV (SCCL-5) and 127.6 mV (SCCL-2). The coal samples having WOPD values less than 100 mV were SCCL-1, SCCL-5 and SCCL-6, indicating that they were poorly susceptible to spontaneous heating. The coal samples SCCL-7, SCCL-8, MCL-1, MCL-2, MCL-4, MCL-5, SECL-2, SECL-3, SECL-4, SECL-5 and SECL-6 were moderately susceptible as they had WOPD values between 100-120 mV. The coal samples SCCL-2, SCCL-3, SCCL-4, MCL-3, MCL-7 and SECL-5 were highly susceptible as they had WOPD values greater than 120 mV.

The dependence of spontaneous heating susceptibility of coals on its intrinsic properties was analyzed by correlation studies between them. The summarized values of correlation coefficients between values of correlation coefficients are shown in table 6.1.

Table 6.1: Summarized values of correlation coefficients between susceptibility indices and intrinsic properties

Sl. No.	Susceptibility indices → / Intrinsic Properties ↓	CPT (°C)	DTA				WOPD (mV)
			T_c (°C)	II A slope	II B slope	II slope	
1	M (%)	0.717	0.380	0.804	0.701	0.416	0.087
2	VM (%)	0.127	0.880	0.503	0.870	0.320	0.308
3	A (%)	0.160	0.533	0.462	0.624	0.210	0.394
4	GCV (Kcal/Kg)	0.264	0.586	0.628	0.822	0.143	0.337

From a critical analysis of chapter 5 and table 6.1, the following important observations can be made.

- Earlier work (Gouws and Wade, 1989) suggested characteristic temperature (T_c) as a measure of spontaneous heating susceptibility. On the contrary, average slope of

stage IIA was preferred by Sahu etal. (2005) over T_c. In the present work, the constituents of proximate analysis and GCV show comparatively high correlation with the average slopes of both stages II A and II B than other parameters. So, both these parameters may be taken as a measure of spontaneous heating susceptibility.

- Moisture and CPT values showed a good correlation but other intrinsic properties showed low values. So, CPT may not a reliable method of assessment of spontaneous heating susceptibility.
- The wet oxidation potential differences show correlation values below 0.4 with the intrinsic properties. Therefore, this method may not be a suitable measure of spontaneous heating susceptibility.
- Earlier work (Sahu et al., 2005) showed that proximate analysis parameters showed better correlation with susceptibility indices than ultimate analysis constituents. In this work, it may be observed that among the intrinsic parameters, moisture and volatile matter showed good correlation with the susceptibility indices whereas Ash content and GCV values did not show significant correlation.

6.2 CONCLUSION

From the above discussion, it may be concluded that the coal sample SECL-2 had extreme values in most of the experiments which indicate that it is highly susceptible to spontaneous heating. In actual field conditions, the coal seam had an incubation period of only 35 days which is thus supported by the experimental investigations. Among other samples, SECL-1, SECL-3, MCL-7, SCCL-3, SCCL-4 and ECL-1 may be considered as moderate to highly susceptible coals by analysis from susceptibility indices.

From correlation analysis, it may concluded that average slopes of stage II A and II B together when taken into consideration, may give a fairly accurate measure of spontaneous heating susceptibility as they showed high correlation with the intrinsic properties. Moisture content and volatile matter may also be helpful in assessment of liability of coal to spontaneous heating as they showed better correlation than other intrinsic properties like ash content and gross calorific values.

The empirical relationships established from curves of best fit between the susceptibility indices and intrinsic properties were better and showed higher correlation values when statistical softwares with a wider variety of regression models for curve fitting were used. It was also observed that due to a few extreme values in the correlation plots, the curve fit may be erroneous to some extent and cannot be used as a reliable means for interpolation. A solution to this problem may be use of more number of samples for correlation plots as more the number of samples, better is the curve fit.

The wet oxidation potential difference values showed comparatively poor correlation than DTA parameters. The CPT method may not be used as a conventional method for predicting susceptibility of coals to spontaneous heating as it also showed poor correlation with intrinsic properties which was also found in past work by researchers (Sahu et al. 2005). Moreover, it is a time consuming method having little reproducibility of results. So, it is worth mentioning that differential thermal analysis is a better method for assessment of spontaneous heating susceptibility of coal than crossing point temperature and wet oxidation potential analysis.

CHAPTER 7

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7. REFERENCES

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APPENDIX – I

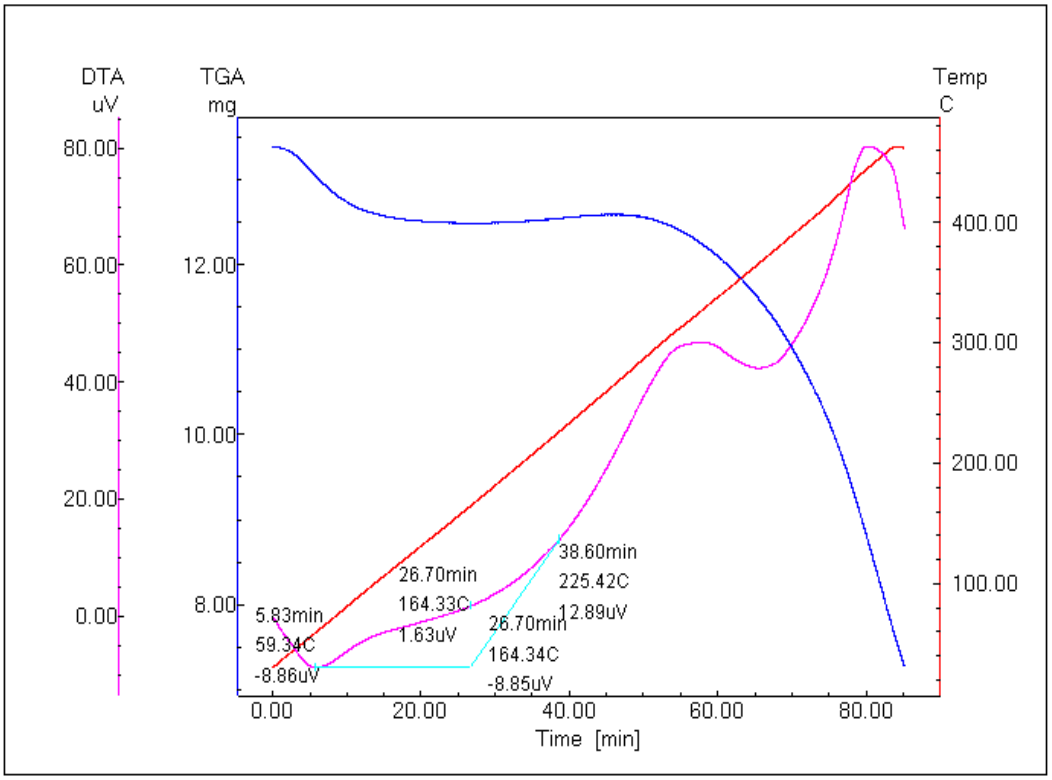
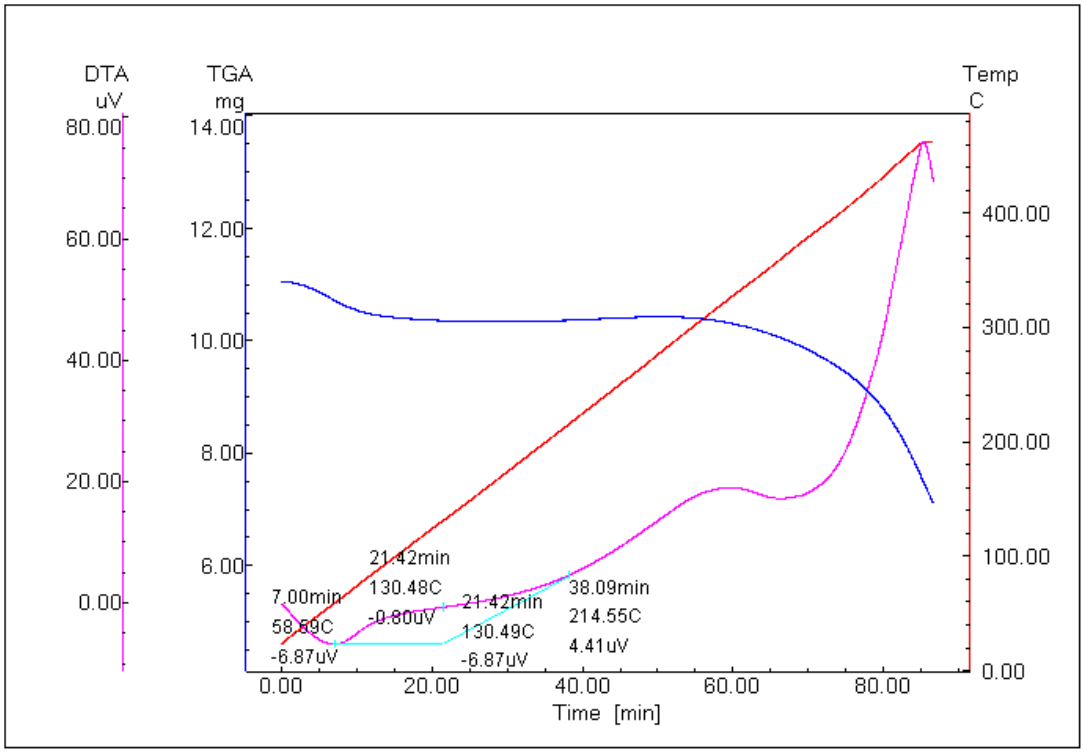


Figure A1.1: DTA thermogram for sample SCCL-1



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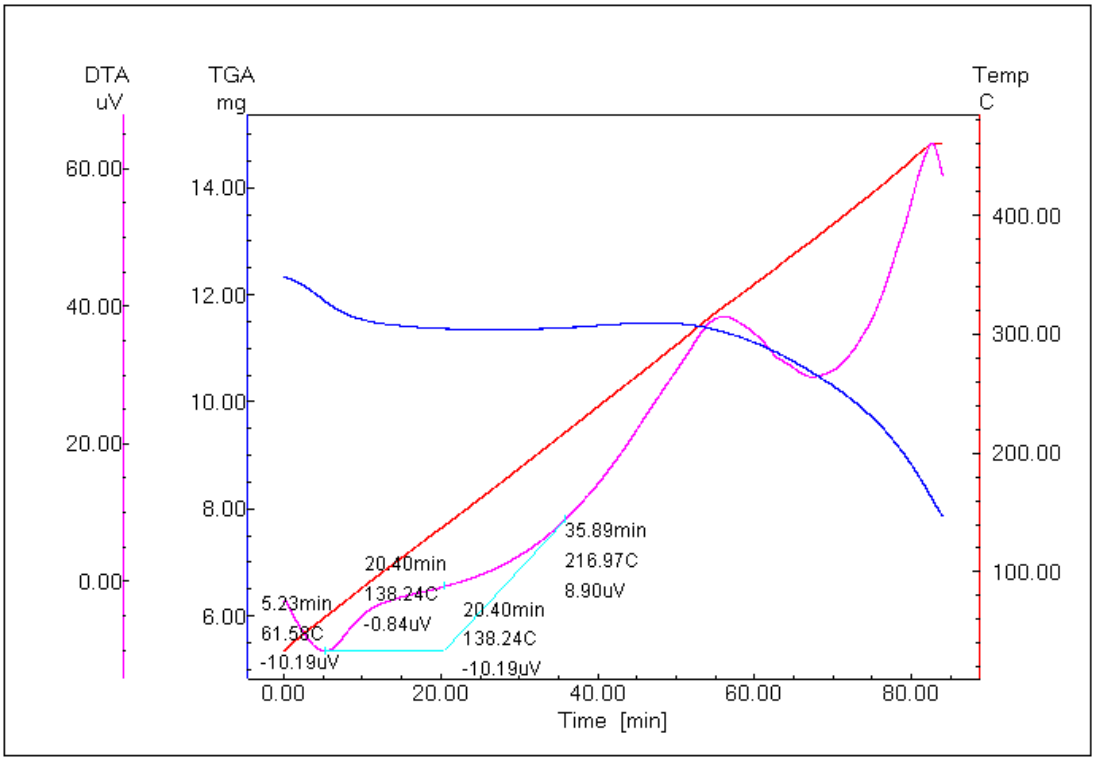

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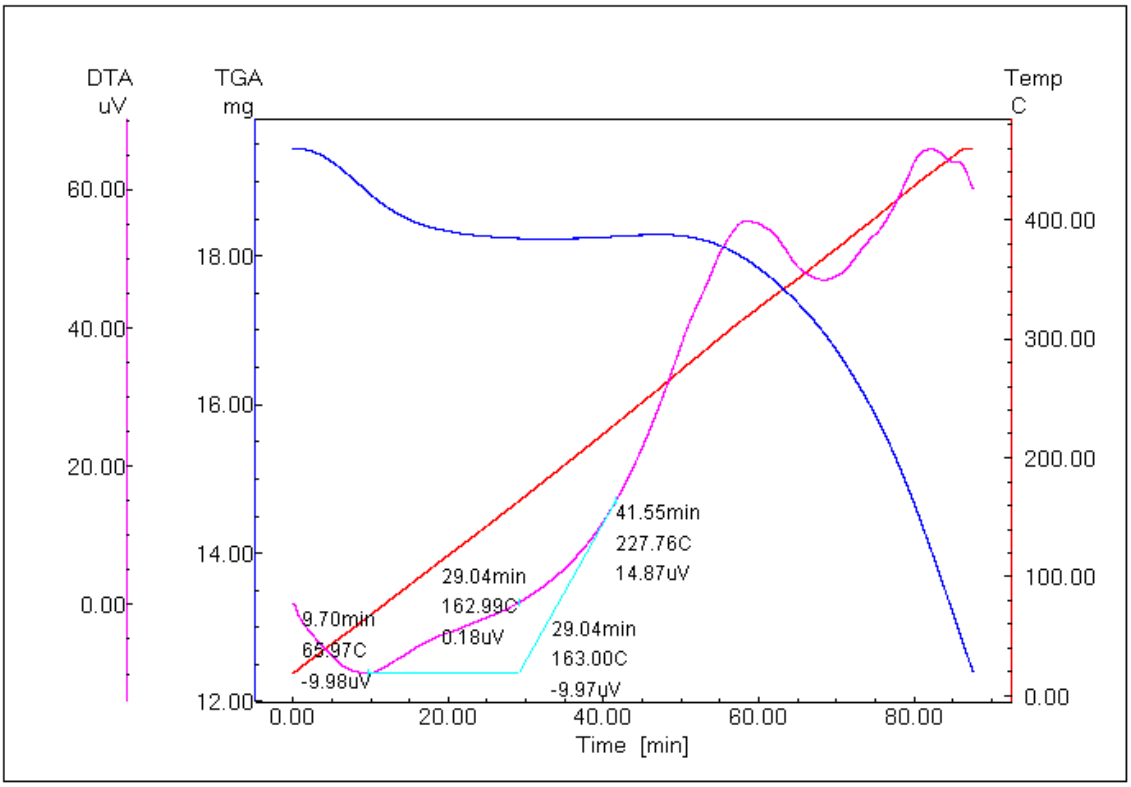


Figure A1.4: DTA thermogram for sample SCCL-4

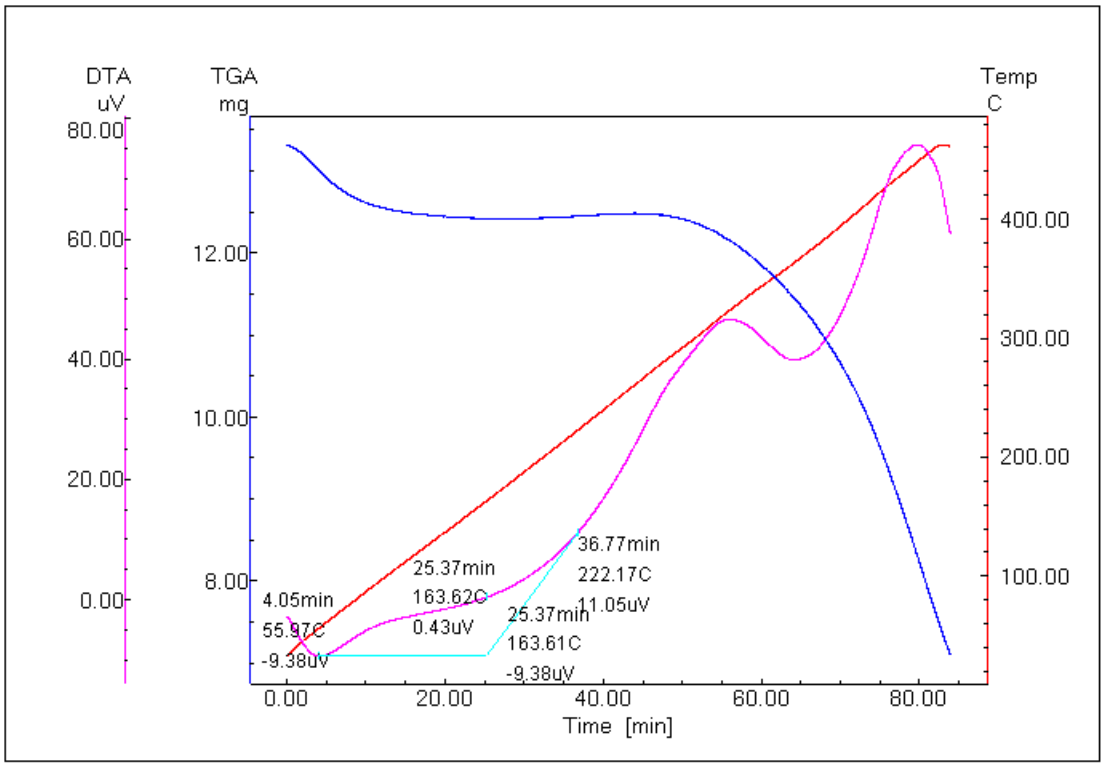


Figure A1.5: DTA thermogram for sample SCCL-5

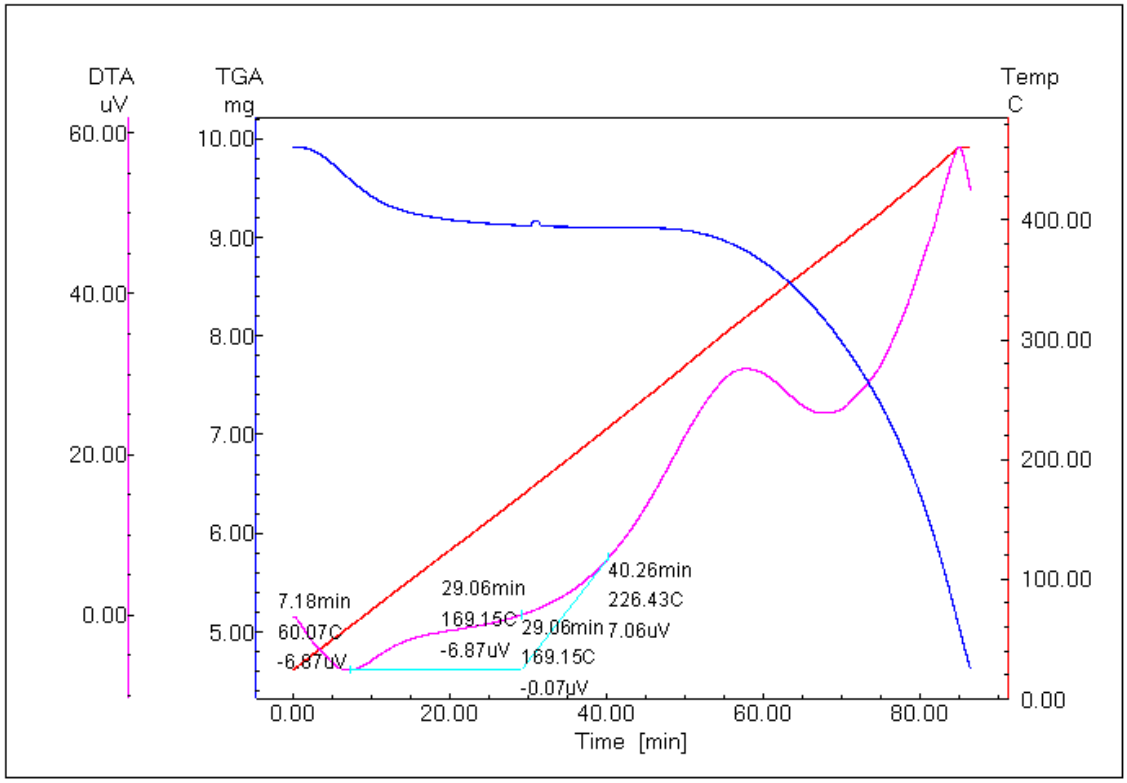


Figure A1.6: DTA thermogram for sample SCCL-6

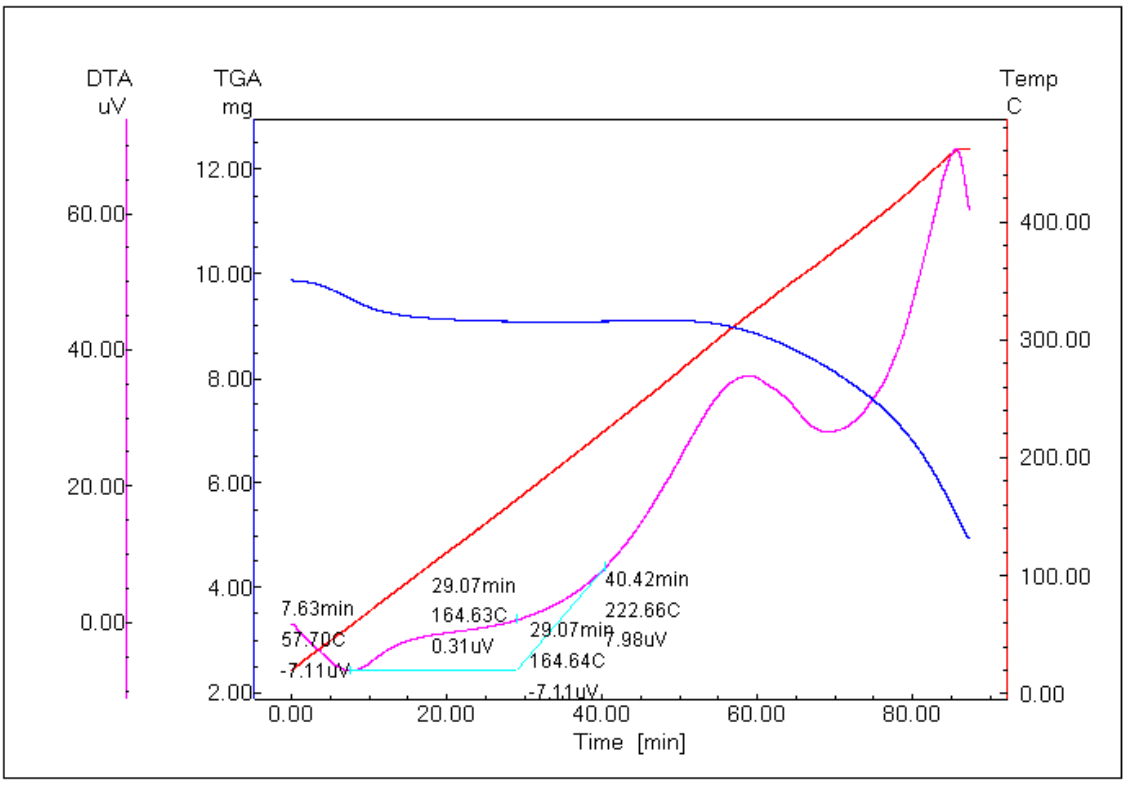


Figure A1.7: DTA thermogram for sample SCCL-7

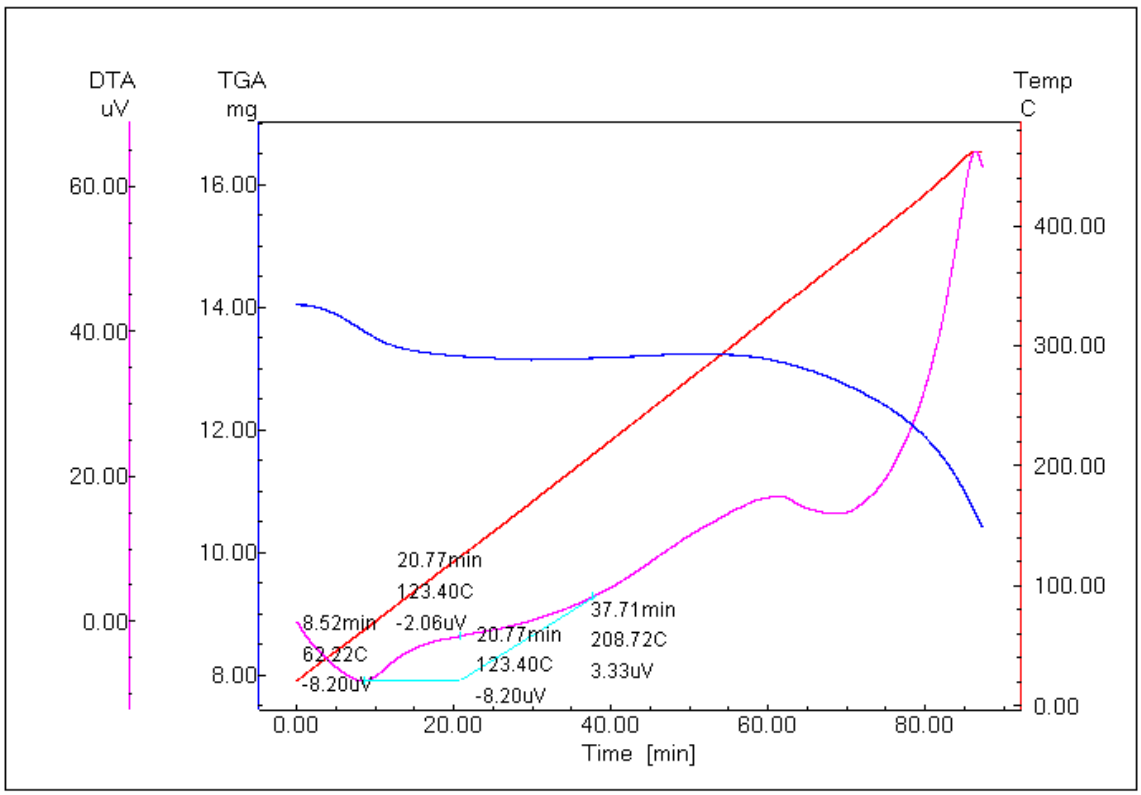
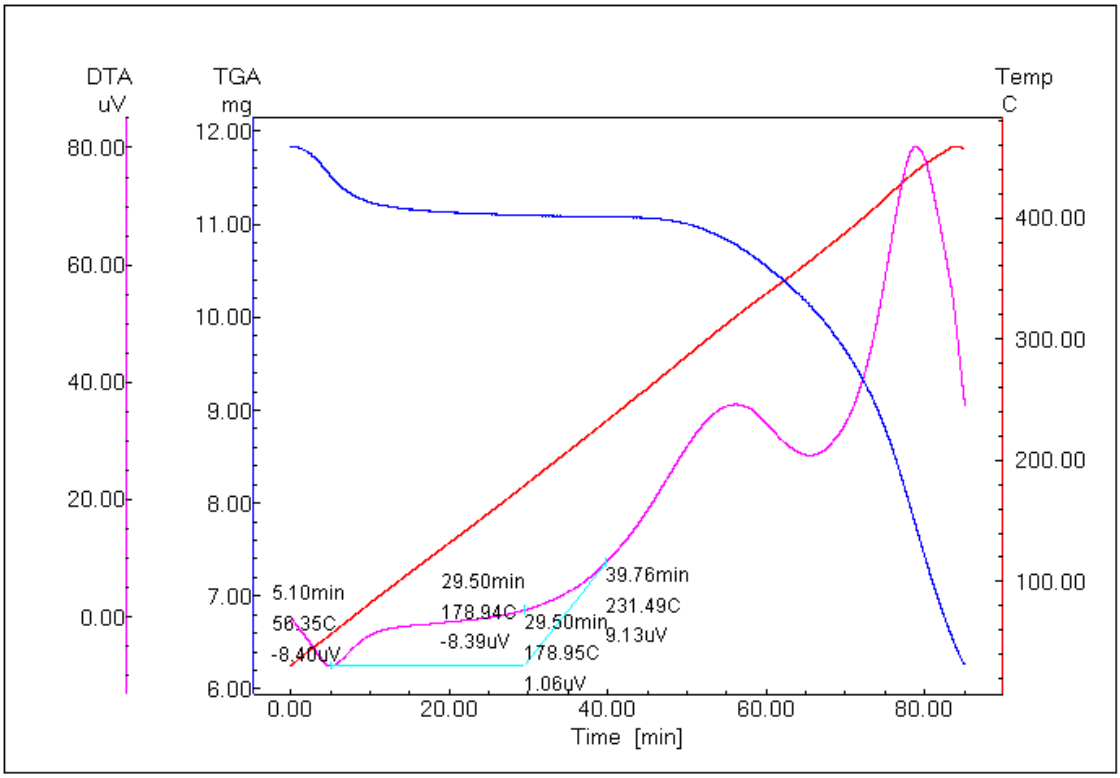


Figure A1.8: DTA thermogram for sample SCCL-8



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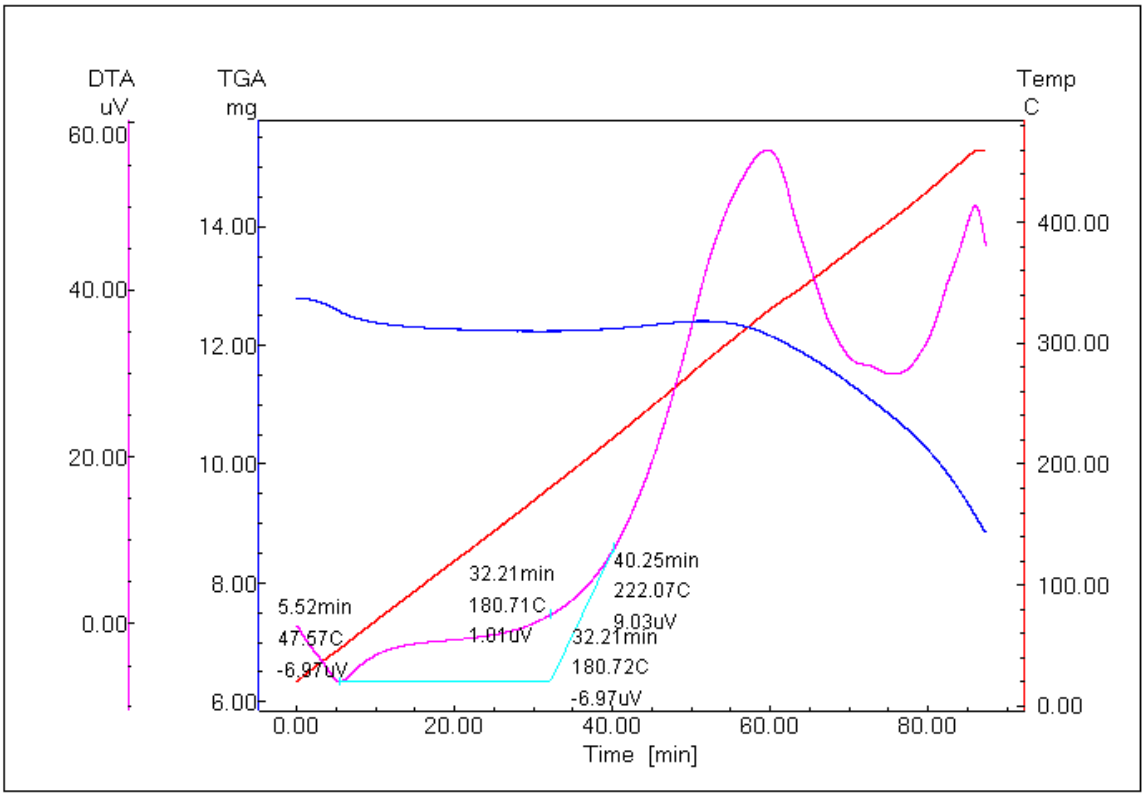

Figure A1.10: DTA thermogram for sample MCL-1

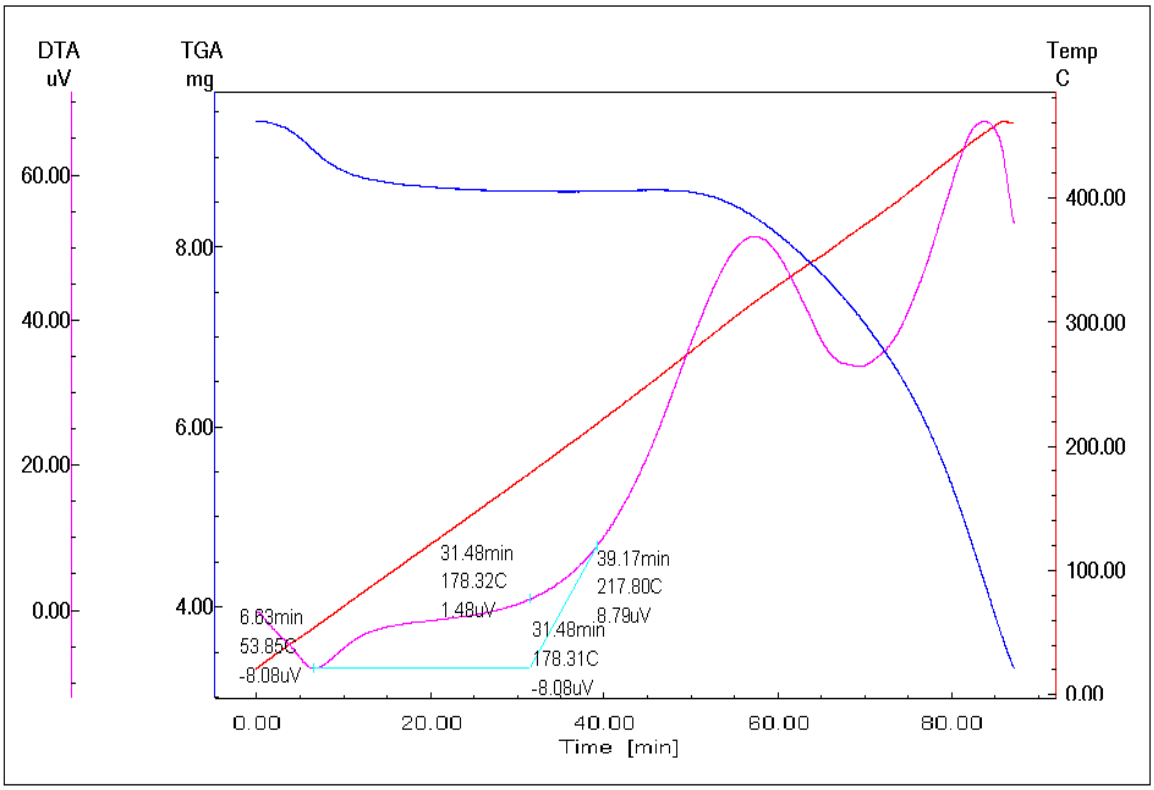

Figure A1.11: DTA thermogram for sample MCL-2

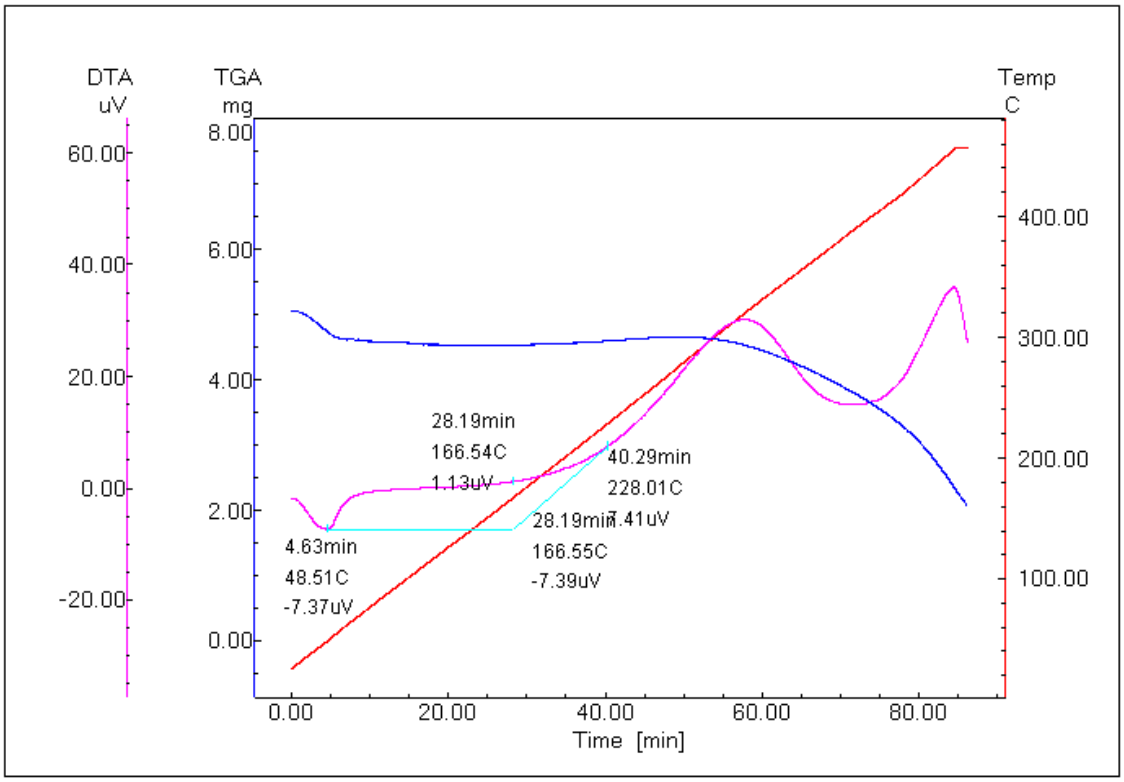

Figure A1.12: DTA thermogram for sample MCL-3

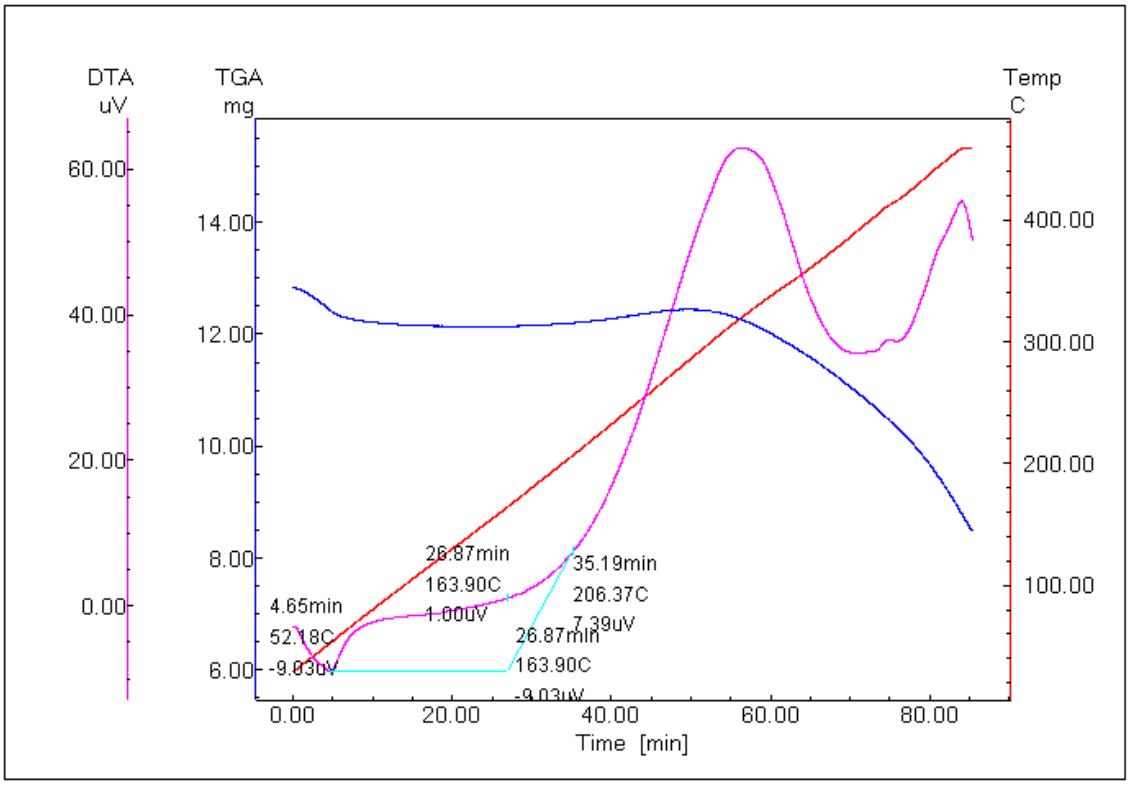

Figure A1.13: DTA thermogram for sample MCL-4

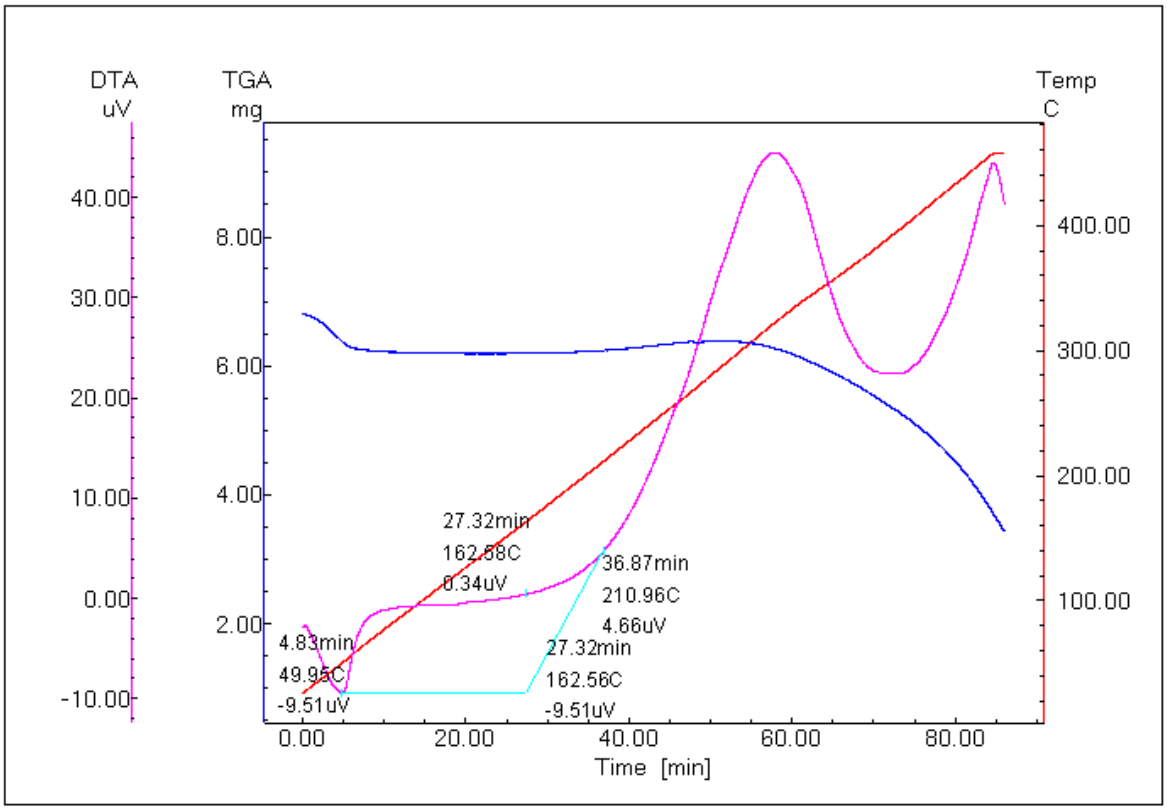

Figure A1.14: DTA thermogram for sample MCL-5

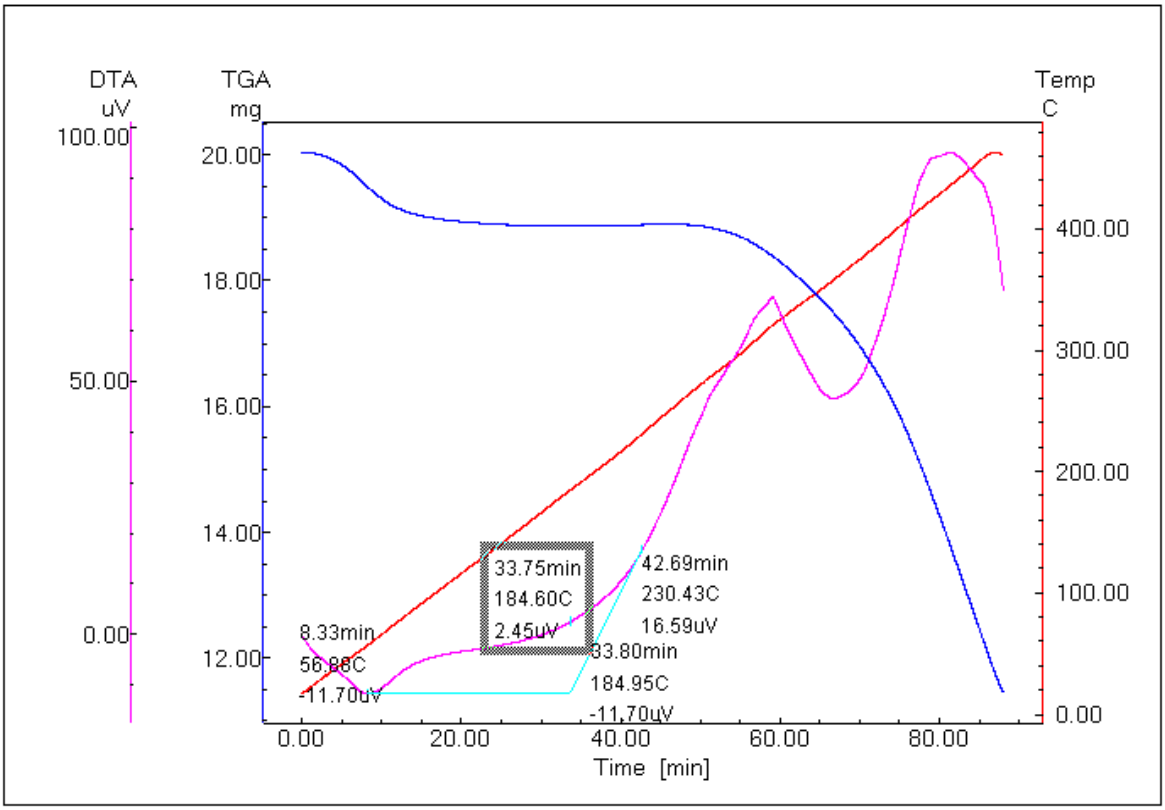

Figure A1.15: DTA thermogram for sample MCL-6

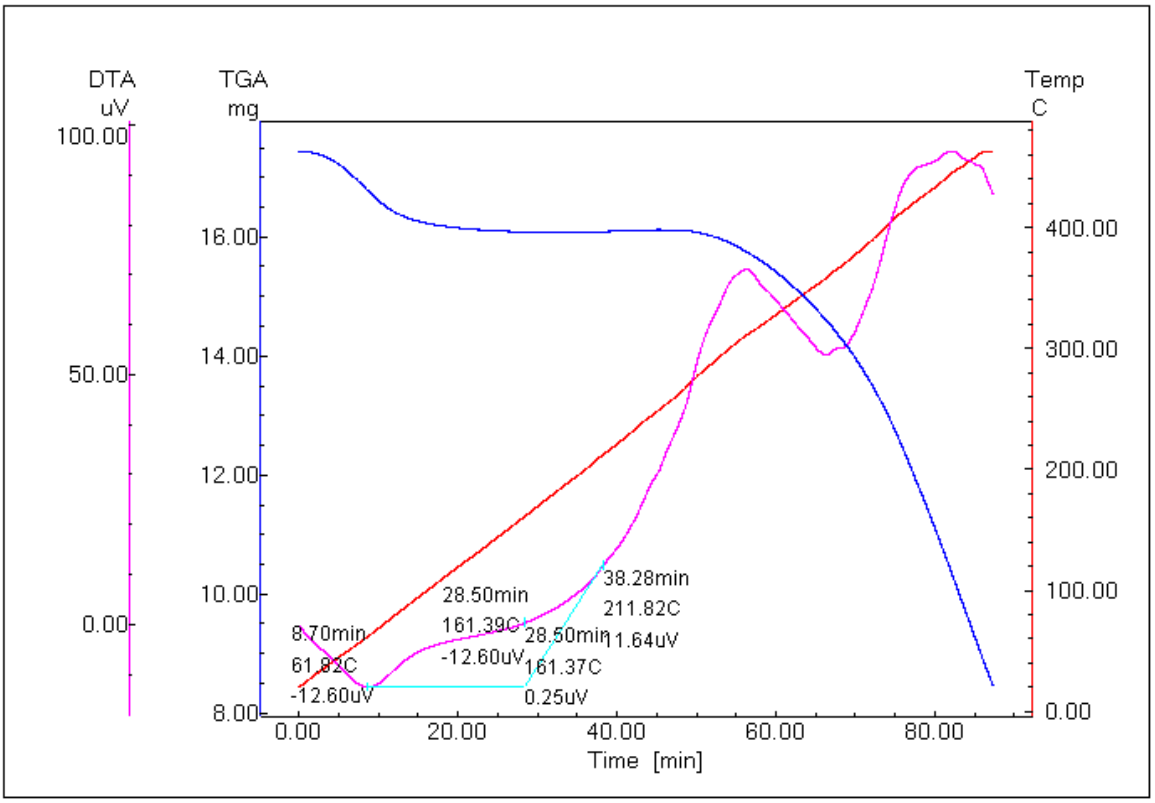

Figure A1.16: DTA thermogram for sample MCL-7

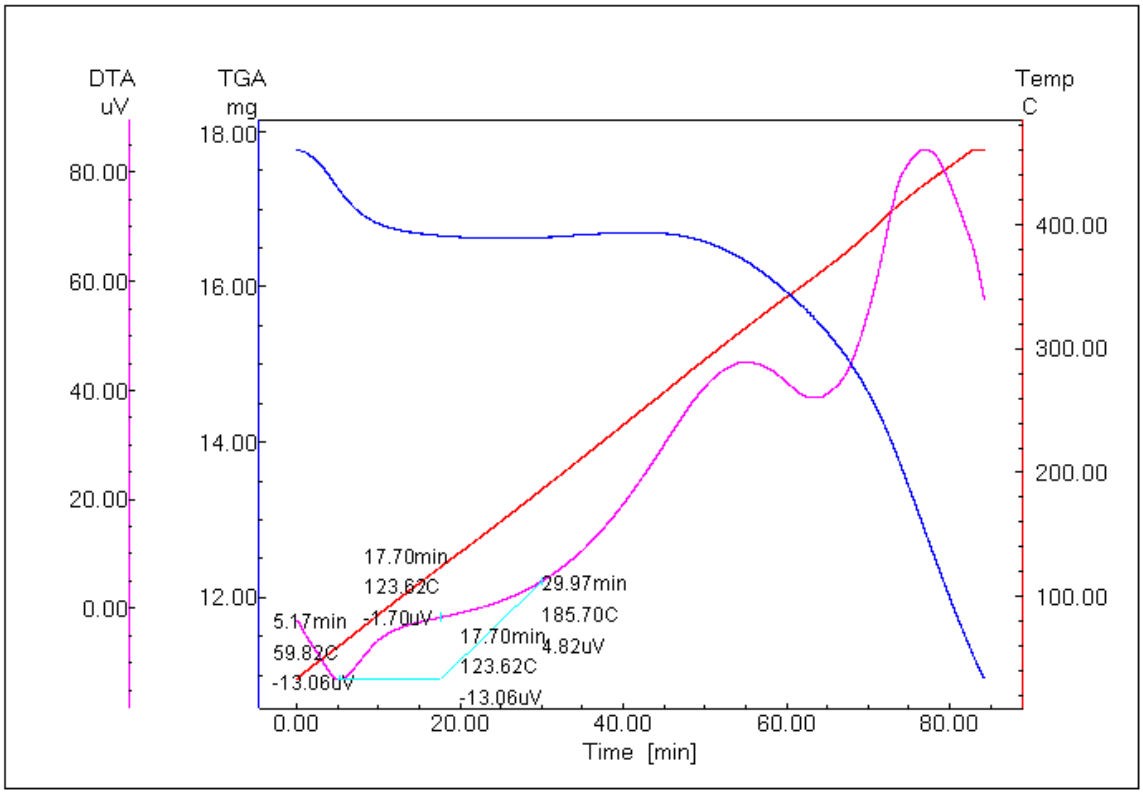

Figure A1.17: DTA thermogram for sample SECL-1

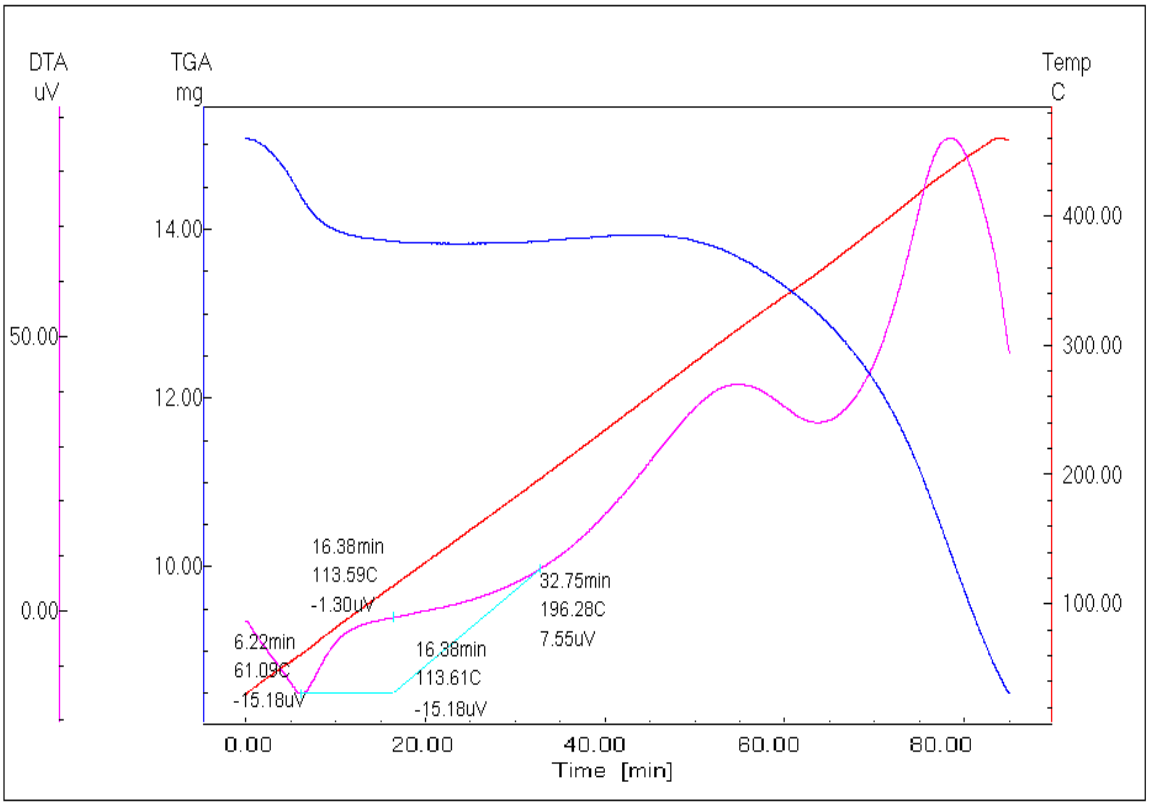

Figure A1.18: DTA thermogram for sample SECL-2

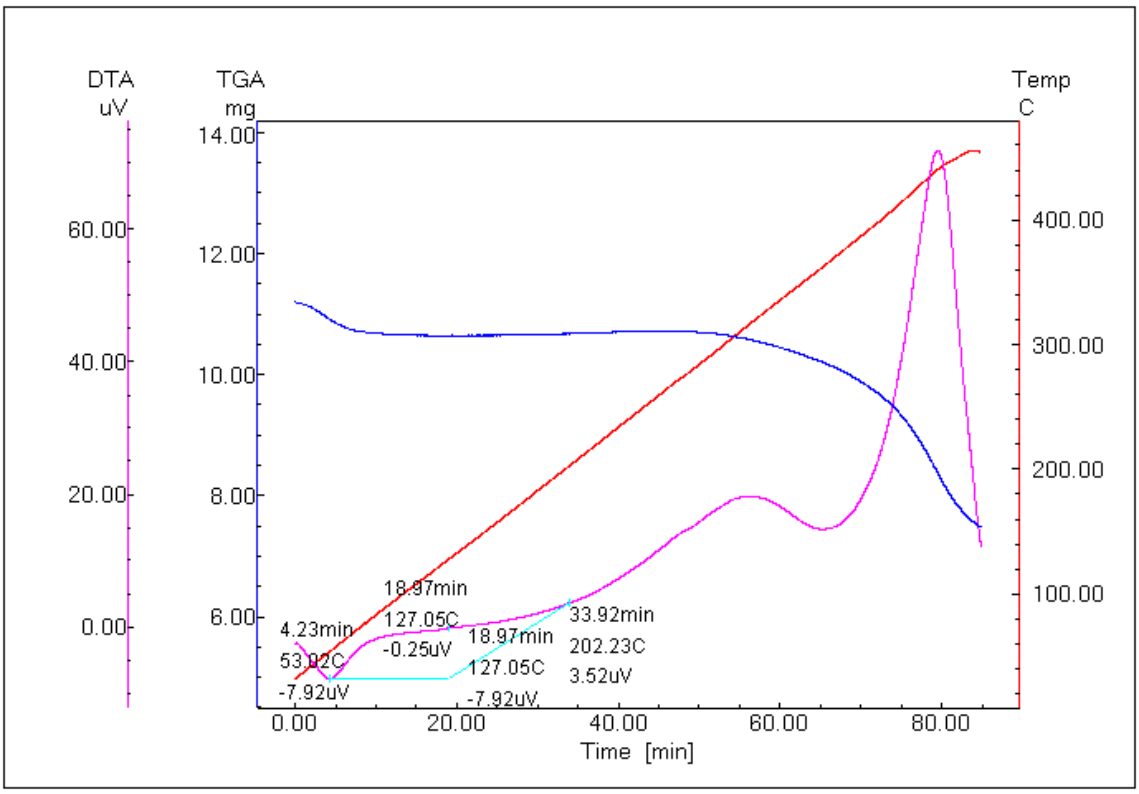

Figure A1.19: DTA thermogram for sample SECL-3

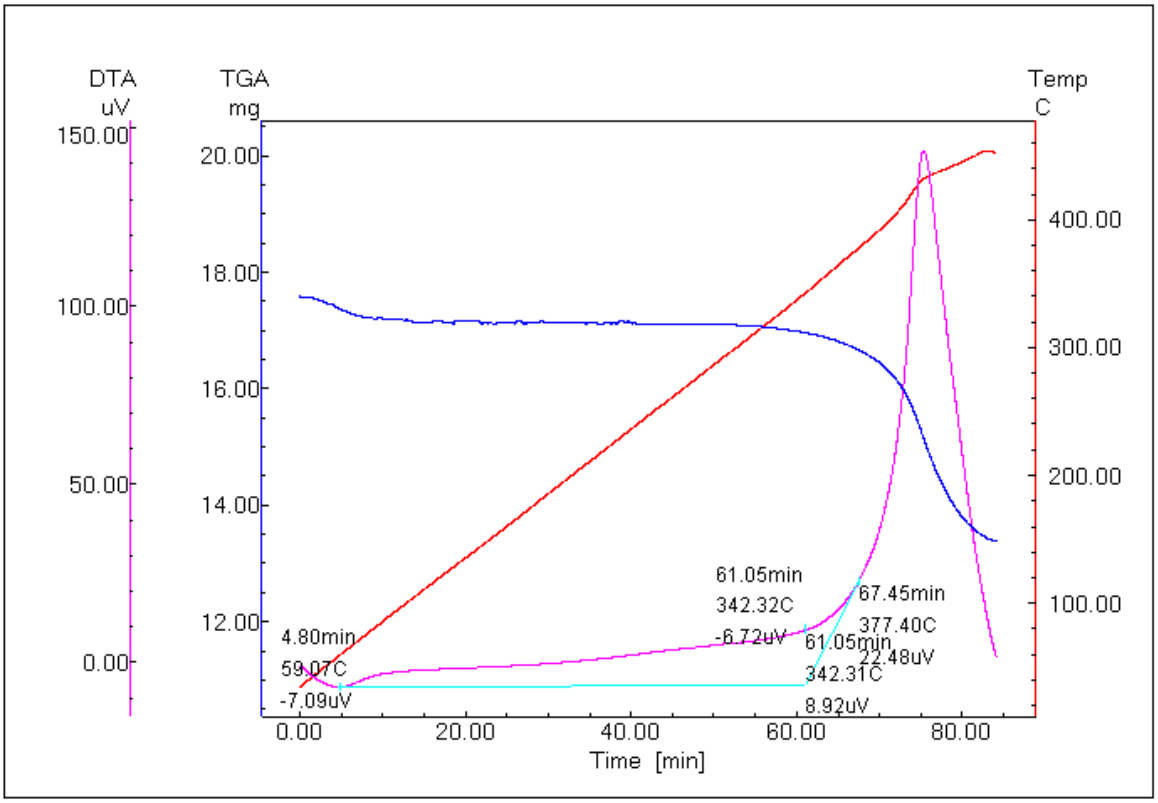

Figure A1.20: DTA thermogram for sample SECL-4

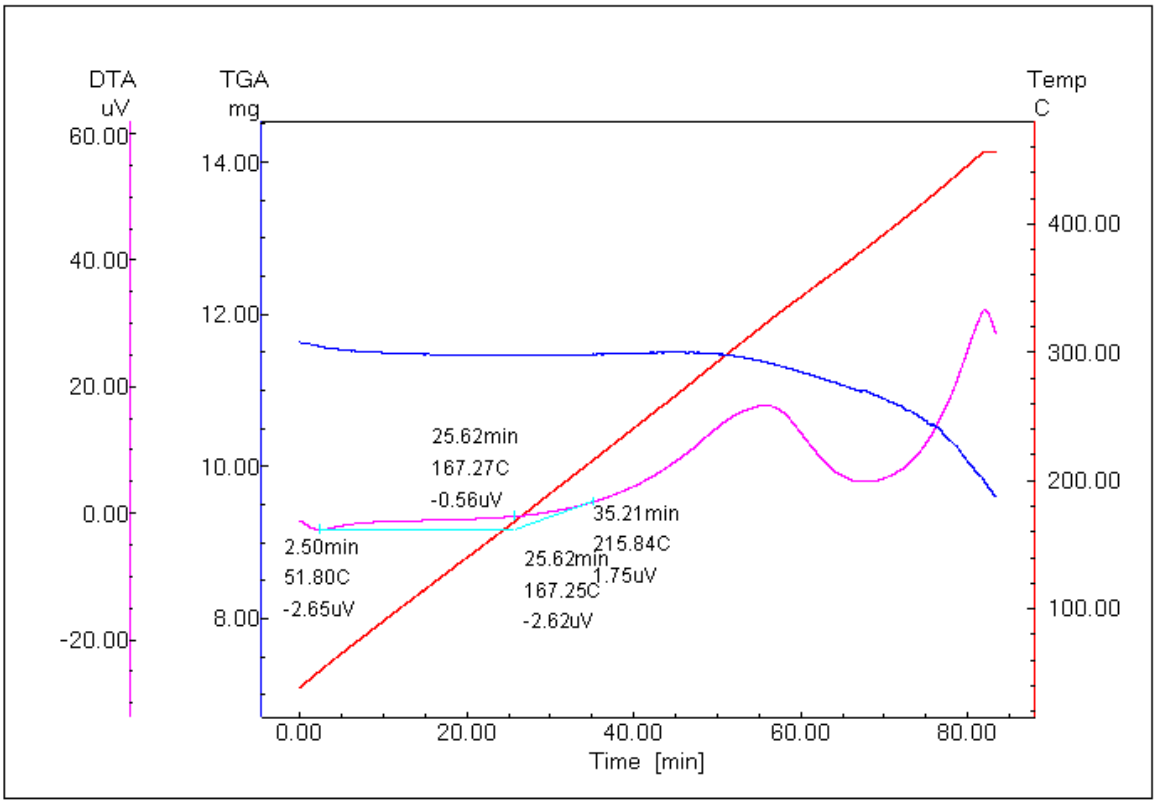

Figure A1.21: DTA thermogram for sample SECL-5

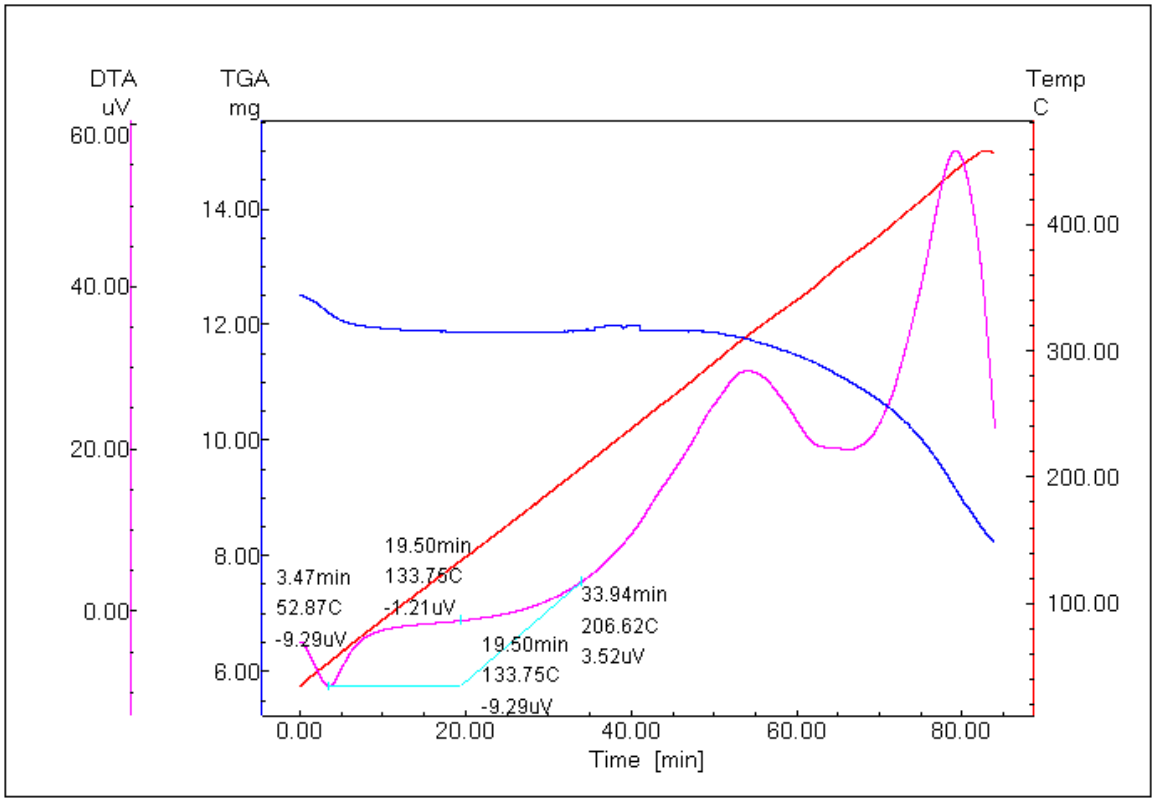

Figure A1.22: DTA thermogram for sample SECL-6

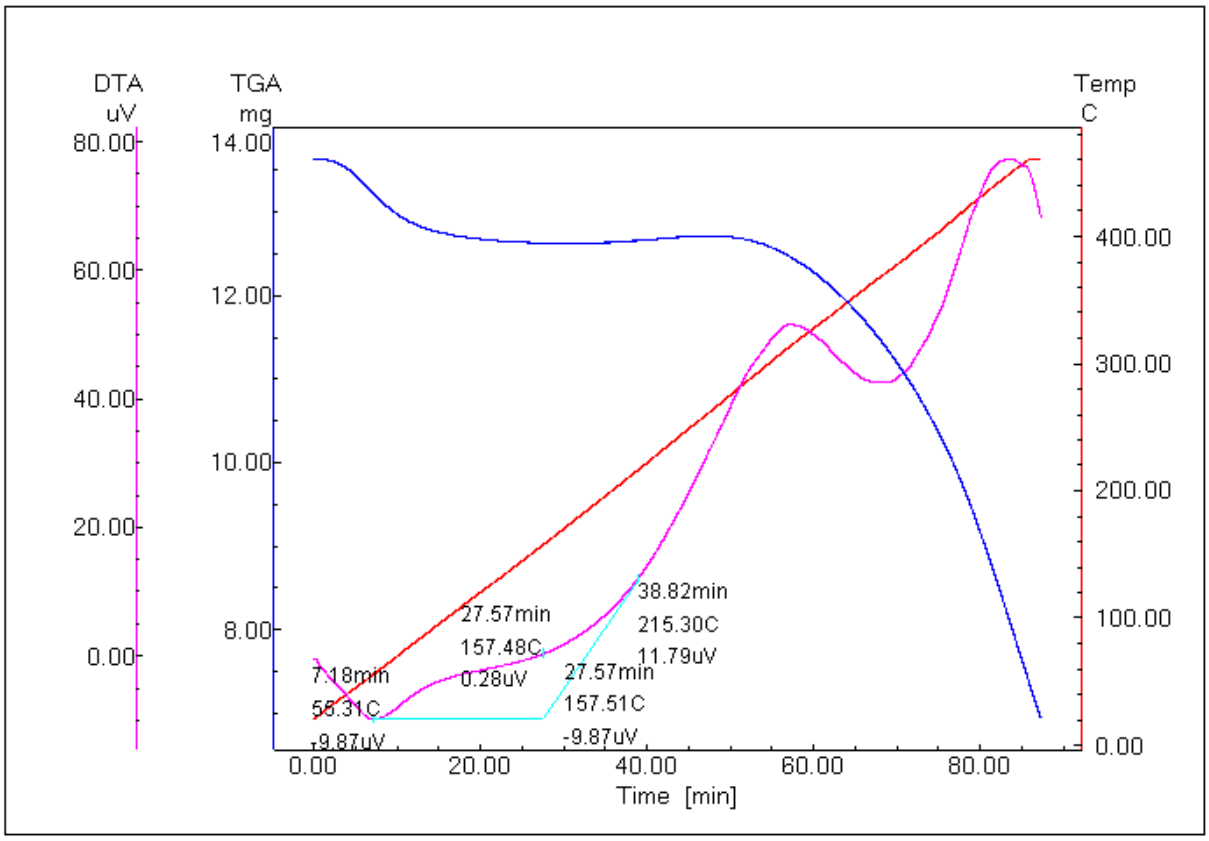

Figure A1.23: DTA thermogram for sample ECL-1

APPENDIX-II

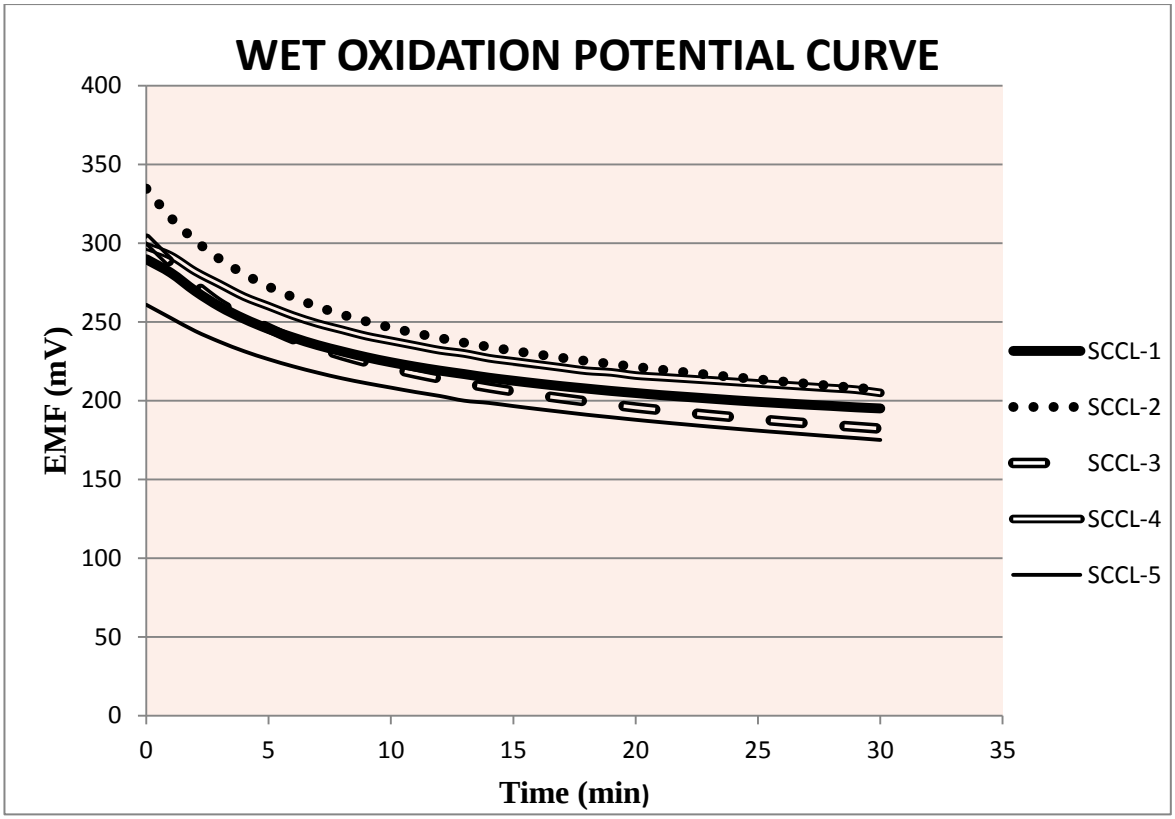

Figure A2.1: Wet Oxidation Potential curves for samples SCCL 1-5

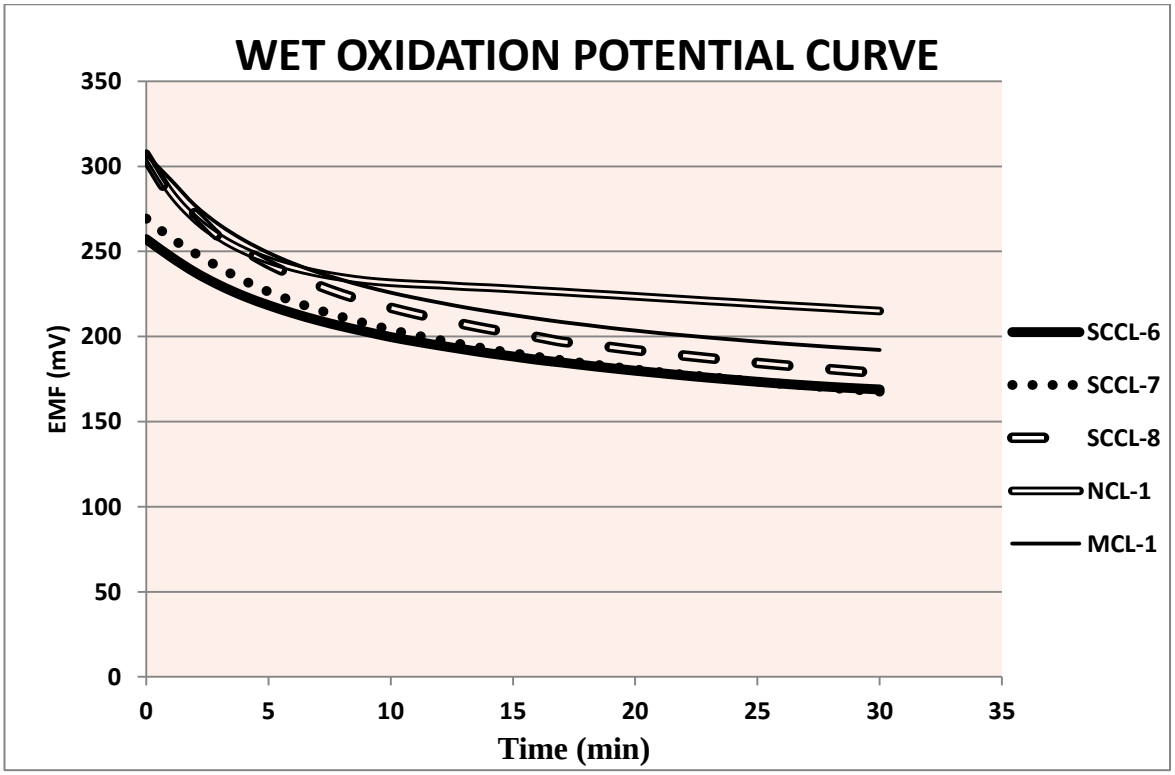

Figure A2.2: Wet Oxidation Potential curves for samples SCCL 6-8, NCL-1, MCL-1

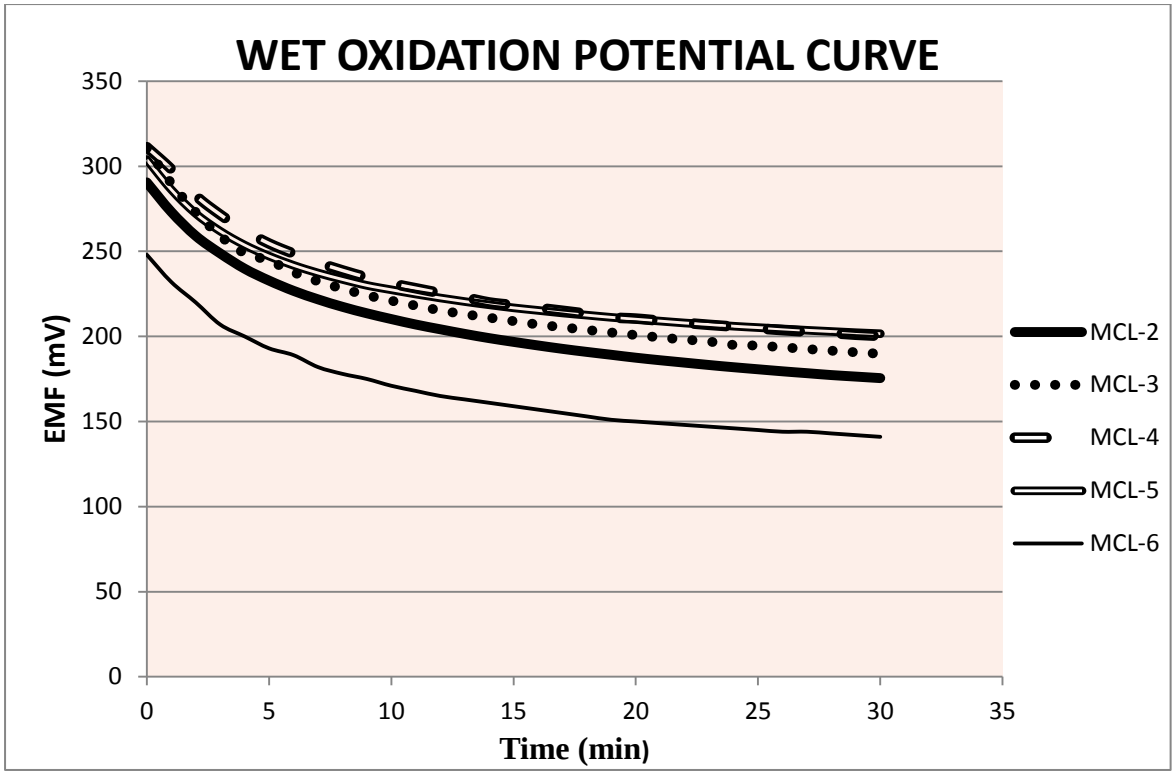

Figure A2.3: Wet Oxidation Potential curves for samples MCL 2-6

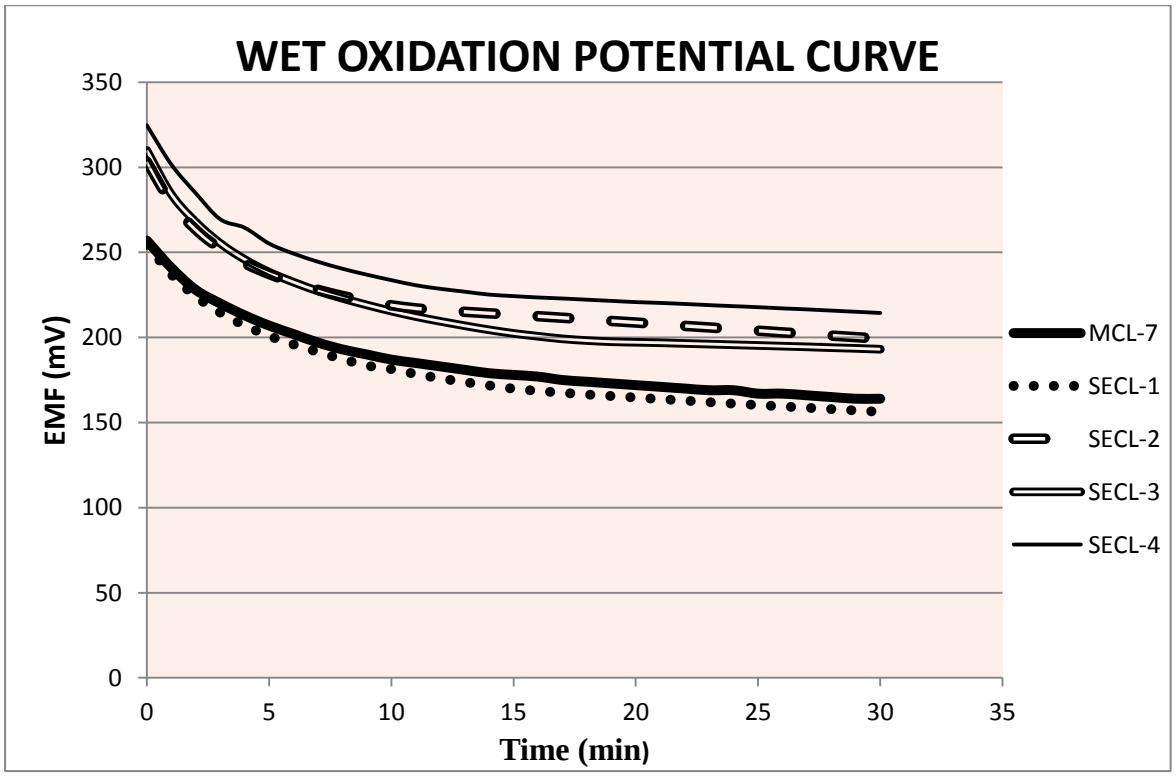

Figure A2.4: Wet Oxidation Potential curves for samples MCL-7, SECL 1-4

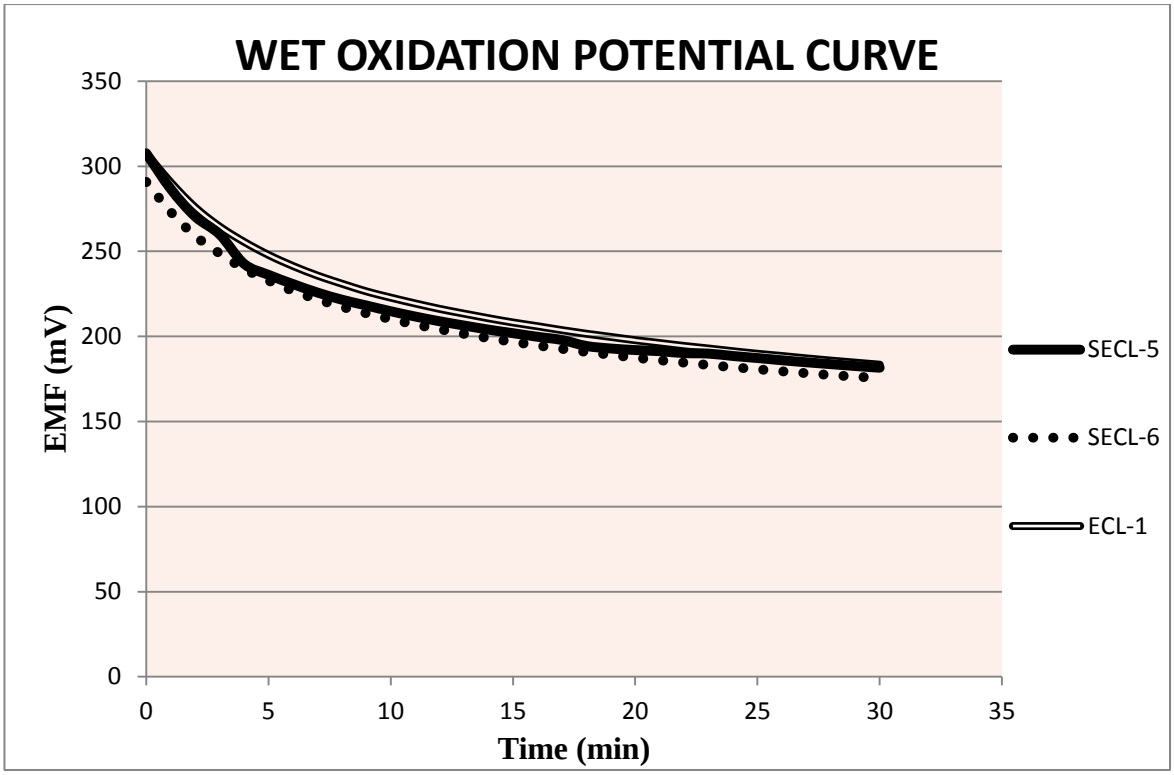

Figure A2.5: Wet Oxidation Potential curves for samples SECL 5-6, ECL-1